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NASA TM X- 63025

COMPILATION, METALLOGRAPHIC AND RELATED METALLURGICAL TESTS

BY
W. G. GRENIER

JANUARY 1966

GODDARD SPACE FLIGHT CENTER
GREENBELT, MD.

COMPILATION, METALLOGRAPHIC AND RELATED METALLURGICAL TESTS

by

W. G. Grenier

January 1966

FOREWORD

A compilation of the Metallurgical and related tests, conducted during the calendar year of 1964, that were conducted by the Structural & Mechanical Applications Section, Mechanical Systems Branch, Spacecraft Integration and Sounding Rockets Division, Technology Directorate, is herein contained.

The principal purpose of this document is to compile under one cover, all Metallurgical and related studies conducted in the calendar year of 1964 by personnel of the Structural and Mechanical Applications Section. The tests have been in direct support of specific spacecraft programs and the A.T.D. effort. The studies consist of: Micro Electronic Circuit Anomalies, Physical Properties of proprietary coatings, and Metallurgical Examinations of specific alloys. In all cases, conclusions made as a result of these tests were those of the Requestor or his consultant.

CONTENTS

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
D. F. Fitzpatrick	11	S-121	AE-B (S-GA)	673S65-01	401-55

DATE IN	DATE COMPLETED	PERFORMED BY
6-11-64	Interim Report 7-21-64	W. G. Grenier and D. F. Freedman

NAME OF TEST

Solar Cell Bonding with Low Temperature Melting, Low Vapor Pressure Alloys.

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Service: Exploration of feasibility of various Gallium alloys as bonding agents for solar cells.

EQUIPMENT INVOLVED:

The metals: Gallium, tin, copper, gold, and 60/40 tin/lead solder. Soldering iron, propane torch, belt surfacer, Handimet hand polisher, 240 grit silicon carbide paper, Ohaus triple beam balance, teflon beakers and stirring rods, brass weight, pruified copper metal foil, 0.030 thick mild steel sheet, 'C' clamps, heat treating furnace, various solvents and other hand tools including Chatillon tensile tester.

RESULTS:

Approach No. 1, Ga-Sn-Cu amalgum, see Table 1, Sheet 5

Approach No. 2, See Sheets 7-9 inclusive

Approach No. 3, Alloys used for experiment, Table 2, Sheet 12
Lap joint bond strength, Table 3, Sheets 15 and 16.

W. G. Grenier 7-21-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F.F itzpatrick	AE-B (S-GA)	673S65-01	401-55

PROCEDURE: PREPARATION OF GALLIUM-TIN EUTECTIC

It was desired by the Originator that a quantity of Ga-Sn eutectic alloy be prepared. This was defined as a 90w/o Ga + 10 w/o Sn mixture. Gallium, 99.9999%, was combined with 30 mesh, granular Sn of 99.9% assay, in the prescribed proportions. The coarse granular tin was used in preference to tin powder as per N.B.S. Technical Note 140, footnote 7, page 3.

One hundred grams of the eutectic mixture was prepared and will be stored in a bottle lined with paraffin, prepared expressly for that purpose. The Ga and Sn was blended in a Teflon beaker using a Teflon stirring rod and policeman. Subsequent to blending, the eutectic will be held overnight to assure complete solution of the tin.

W. G. Grenier 6-18-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-01	401-55

PROCEDURE: GENERAL – PRELIMINARY APPROACH NO. 1

Prepare 10 gms Cu + Eutectic alloy;

Ga-Sn : 3.4 gms

Cu (powder) : 6.6 gms

As a Teflon mortar and pestle is not available this amalgum will be blended in a small glass beaker.

Beaker wt = 47.2 gms

add 6.6 gms Cu = 53.8 gms

add 3.4 gms Ga-Sn = 57.2 gms total

Ga-Sn eutectic wet sides and bottom of beaker. Therefore not all of the eutectic was available to the copper powder. By weighing the wetted beaker after removal of the amalgum it is learned that the Ga-Sn remaining is less than 0.1 gm.

As the Cu-Ga-Sn amalgum began to become unworkable it was removed from the beaker, placed in an envelope fabricated from filter paper, and compacted in a machinists vise. It was removed from the vice, and unwrapped at 0916 hours. At this time it is a small crumbly cake. See Figure 1.

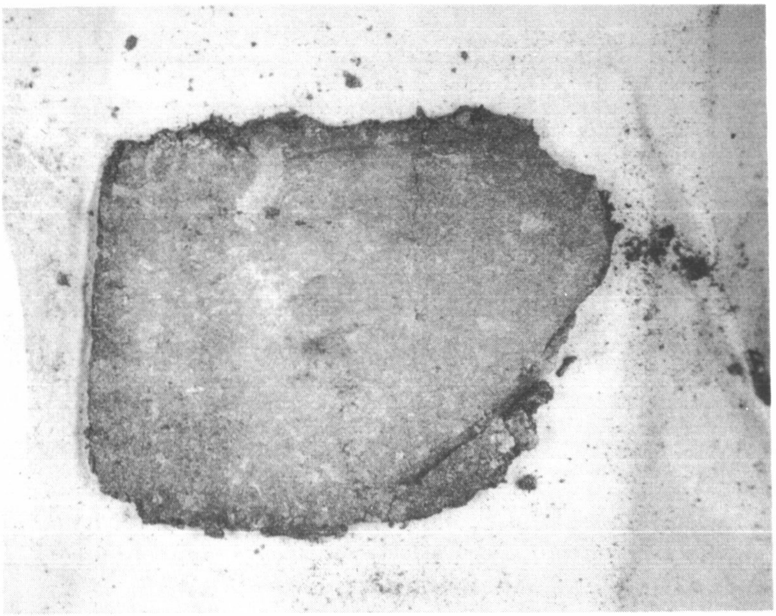
From Table 1 of N.B.S. Technical Note 140, it can be expected that the set-up time for this mixture will be approximately 4 hours. Rudimentary tests for crumbling will be conducted every hour on the hour beginning at 1000 hours. Set-up will be considered complete when cake no longer crumbles readily.

W. G. Grenier (Signature) 6-19-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-01	401-55

DATA: GENERAL - PRELIMINARY APPROACH NO. 1



1-2/3X

Figure 1—Appearance of Amalgum Cake After Removal From the Vise.

670-18 (2/64)

W. G. Grenier (Signature) 6-19-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-01	401-55

DATA:

Table 1
SET-UP TIME FOR Ga-Sn-Cu ALLOY

TIME (HOURS)	CONDITION
0916	Compacted—very crumbly—fractures easily
1000	very crumbly
1100	very crumbly
1200	Appreciably harder—still crumbly
1300	very crumbly
1400	very crubmly, still no strength
1500	While material appears to be harder, it still crumbles readily
1600	Same as at 1500 hours
1700	Still crumbly.

This is already far in excess of the expected 4 hours.

Note: Specimen checked at 0916 on 6-23-64, 72 hours set = weak and brittle < R/B-0
but not crumbly.

670-19 (2/64)

W. G. Grenier (Signature) 6-19-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-01	401-55

PROCEDURE: GENERAL – APPROACH NO. 2

It was noted that the "Wet Cement" viscosity characteristics of the Ga-Sn-Cu amalgum used, formed a good packing type material, but had very little fluidity. That lack of fluidity tends to preclude this particular alloy from use as a cold solder type material. Further, on careful examination of the literature, the weakness and low hardness is readily explained as a result of no compacting pressures. These properties were fully discussed with the Originator and it was determined to try another approach.

Several strips of sheet steel were prepared for use. Each strip was nominally 0.030" thick × 1/2" wide × 5-3/16" in length. The surfaces of each strip were carefully cleaned by abrading on the wet belt surfacer and by hand, using a 240 grit wettable silicon carbide paper. Each strip was then rinsed in free flowing water, followed immediately by a brief immersion in Propanol and dried by an air blast. All surfaces were promptly wiped with a light lubricating oil, to prevent corrosion.

Purified Cu metal foil, 0.002" thick, was supplied by the Originator. This was cut into small one centimeter squares, to be used as alloying shims. Four steel strips were degreased in trichloroethane and tinned with a 60% Sn + 40% Pb solder, to a distance of approximately 2 inches from one end, on one surface. Two of the one centimeter square pieces of Cu shim material were cleaned in hot Triton-X-100 detergent, rinsed in Propanol, and air dried. The tinned areas of the steel strips, were carefully wetted with the Ga-Sn

W. G. Grenier (Signature) 7-6-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-01	401-55

PROCEDURE: GENERAL – APPROACH NO. 2

eutectic of sheet No. 2. A Cu shim was placed over the gallium on one strip of steel and the other strip placed over the Cu, as shown in the schematic diagram of Figure 2, sheet 8. Two samples were prepared in this manner, each being clamped at that location designated by the x. Ordinary 'C' clamps were used with an indeterminant pressure applied.

After twenty-four hours, at room temperature, no cohesion was discernible. On removal of 'C' clamp, the strips fell apart with no external force required. The literature seems to indicate that, while alloying may take place eventually at room temperature, all experiments reported were based on pressure and temperature. Pressures required were not discussed quantitatively, however the temperature used was 150°C and time discussed was 24 hours at that temperature. Therefore reclamped specimens were placed in the heat treating furnace to be held at 125-150°C. They will be checked at the end of four hours.

After four hours, at the indicated temperature of 150°C, no strength was detectable in the joint of one sample examined. That specimen was reclamped and replaced in the oven. Both specimens were held at temperature for 24 hours total time. While some adhesion appeared to exist, it was too slight to measure.

W. G. Grenier 7-8-64
(Signature) *(Date)*

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-01	401-55

DATA: Schematic for Approach No. 2

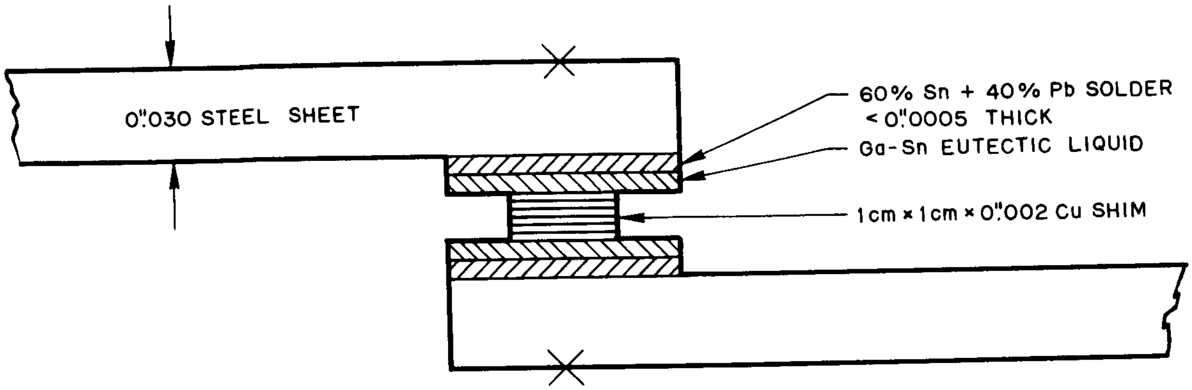


Figure 2–Schematic Representation of Approach as Described on Sheet 7.

670-18 (2/64)

W. G. Grenier — 7-6-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-02	401-55

PROCEDURE: APPROACH NO. 3

Subsequent to Approach No. 2, and in discussion with the Originator it was determined to approach the problem from a different angle. It was determined that several different alloy compositions be evaluated as a preliminary step. Test specimens were prepared as described in Figure 3, sheet 11 of this report. Alloy compositions were prepared in accordance with Table 2, sheets 12 and 13.

As these specimens were prepared at room temperature and under relatively light restrictive load, they were permitted to stand as shown in Figure 4 for 120 hours. At the end of that time the shear strength of the resultant joint was tested by applying a tensile load to the ends of the specimen. A chatillon Model D.P.P. guage and tensile testing stand was used to determine the mechanical strengths of the joints. All tests were conducted at room temperature.

W. G. Grenier 7-15-64
(Signature) (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-02	401-55

DATA: Approach No. 3, 1/2 of Test Specimen

Basis Mtl: 0.030 thick sheet steel.

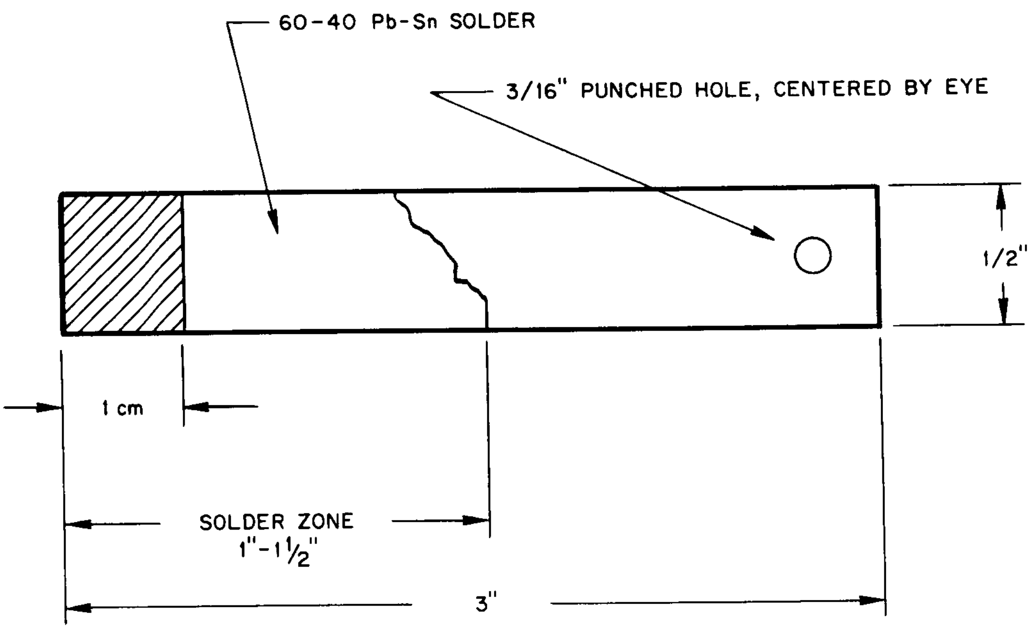


Figure 3

Shows 1/2 of typical lap joint tensile test specimen used in evaluation of specific Gallium alloys. Shading indicates area of lap joint, equal to 0.20 square inches.

A line was scribed, in the solder tinned zone, one centimeter from the end of the 1/2 test specimen. That area only will be wetted with the specified Gallium mixtures.

670-18 (2/64)

W. G. Grenier (Signature) 7-13-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-02	401-55

DATA:

Table 2
SPECIMEN AND ALLOY DESIGNATIONS, APPROACH NO. 3

SPECIMEN NO.	ALLOY NO.	GA-SN EUTECTIC W/O	CU W/O	AU W/O	SN W/O	REMARKS
1	1	80	20	–	–	Wet Silvery alloy,
2	1	80	20	–	–	very fluid.
3	2	70	30	–	–	Not so wet, Silvery loose
4	2	70	30	–	–	amalgum, moderately fluid.
5	3	60	40	–	–	Firm, Silvery, difficult to
6	3	60	40	–	–	wet specimen, no fluidity.
7	4	50	50	–	–	Like dry dental amalgum, Silvery
8	4	50	50	–	–	with Cu flecks, makes thick joint
9	5	80	–	20	–	Very wet, Silvery, no grains of
10	5	80	–	20	–	Cu apparent. Very fluid.
11	6	70	–	30	–	Very wet, Silvery, some Cu grains,
12	6	70	–	30	–	very fluid.
13	7	60	–	40	–	Very wet but better than Alloy 6.
14	7	60	–	40	–	Silvery, very fluid.

670-19 (2/64)

W. G. Grenier 7-15-64
(Signature) *(Date)*

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-02	401-55

DATA:

Table 2
SPECIMEN AND ALLOY DESIGNATIONS (Continued)

SPECIMEN NO.	ALLOY NO.	GA-SN EUTECTIC W/O	CU W/O	AU W/O	SN W/O	REMARKS
15	8	50	–	50	–	Thick and grainy, Silvery, wets
15	8	50	–	50	–	specimen easily, moderate fluidity.
17	9	70	20	10	–	Thick, grainy paste, Silvery, wets
18	9	70	20	10	–	easily, limited fluidity.
19	10	60	30	10	–	Like wet cement, Silvery, wet
20	10	60	30	10	–	specimens by abrasion, no fluidity.
21	11	80	10	–	10	*Very wet, Silvery, large grains of
22	11	80	10	–	10	Sn create difficulties in mating
						surfaces.

*Note: Twenty-four hours later the lapped surfaces of both specimens had mated, as a result of the Sn granuals alloying with the Ga and Cu.

670-19 (2/64)

W. G. Grenier (Signature) 7-16-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-02	401-55

DATA: Approach No. 3, Specimen Preparation

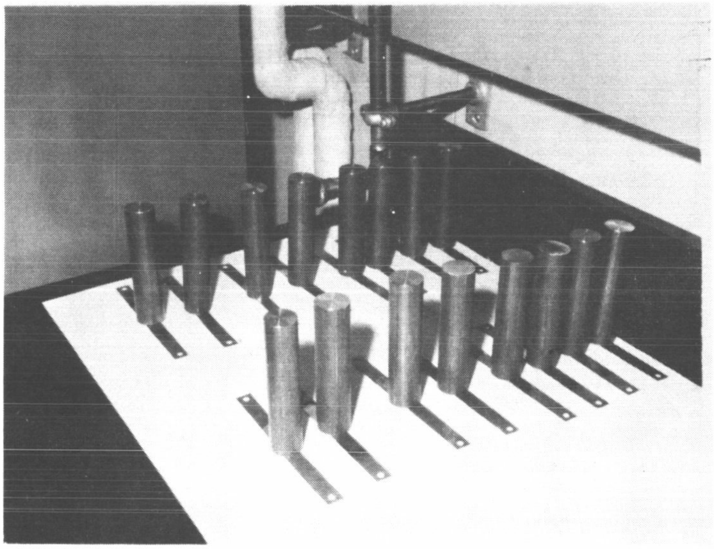


Figure 4–Photograph of Lap Joint Specimens Being Prepared With Different Gallium Alloys.

Brass rods, employed as compressive weights on the joint, each had a mass of 464 grams ± 1/2 gram. Two specimens were prepared for each alloy in this preliminary experiment. Specimens to be held under this pressure and at room temperature for 120 hours.

670-18 (2/64)

W. G. Grenier (Signature) 7-16-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-02	401-55

DATA:

Table 3
ALLOY STRENGTH EVALUATION, UNDER STATIC CONDITIONS

ALLOY NO.	SPECIMEN NO.	TIME SET UP AS FIGURE 3 DATE	TIME SET UP AS FIGURE 3 HOUR	TIME OF STRENGTH TEST DATE	TIME OF STRENGTH TEST HOUR	JOINT STRENGTH LBS.	JOINT STRENGTH P.S.I.	REMARKS
1	1	7-14-64	0935	7-20-64	0932	6.0	30	Joint still wet but pasty
1	2		0935		0935	7.1	35-1/2	Joint still wet but pasty
2	3		1008		1008	8.7	43-1/2	Joint mtl stiff but wet
2	4		1008		1008	5.1	*25-1/2	Joint mtl stiff but wet
3	5		1030		1029	18.5	93	Prestressed to 50 psi Joint is dry and hard
3	6		1030		1040	28	140	Prestressed to 125 psi Joint dry & hard with Cu Pocket in
4	7		1052		1045	19.5	97-1/2	Dry joint–bulky
4	8		1052		1047	20.5	103	Dry joint–bulky
5	9		1113		1112	0.00	0.00	Very wet
5	10		1113		1114	0.00	0.00	Very wet
6	11		1306		1310	0.00	0.00	Wet Paste
6	12		1306		1310	0.1	0	Str. from adhesion paste
7	13		1317		1314	9.7	49	Almost fully dry, stiff and crumbly
7	14		1317		1318	16.0	80	Dry–crumbly

670-19 (2/64)

W. G. Grenier (Signature) 7-20-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-02	401-55

DATA:

Table 3
ALLOY STRENGTH EVALUATION (Continued)

ALLOY NO.	SPECIMEN NO.	TIME SET UP AS FIGURE 3		TIME OF STRENGTH TEST		JOINT STRENGTH		REMARKS
		DATE	HOUR	DATE	HOUR	LBS.	P.S.I.	
8	15	7-14-64	1330	7-20-64	1335	26	130	Prestressed to 125 psi Dry joint
8	16		1330		1335	31-1/2	157	Joint looked good
9	17		1345		1345	12	60	Amalgum broke loose from solder–no apparent alloy
9	18		1345		1345	28-1/2	143	Similar to above
10	19		1355		1358	31-1/2	157	Dry joint but thick
10	20		1355		1360	18-1/2	93	Dry joint but thick
11	21		1410		1410	0.00	0.00	Alloy = liquid
11	22		1410		1410	0.00	0.00	Alloy = liquid

*Alignment of sample halves poor.

Joint strength determinations were conducted using a Chatillon Model D.P.P. guage and tensile testing stand.

670-19 (2/64)

W. G. Grenier 7-20-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
D. F. Fitzpatrick	AE-B (S-GA)	673S65-02	401-55

PROCEDURE:

The tests performed so far indicate that some minimum strength of bond can be achieved with certain gallium alloys without the use of heat.

Additional tests will be required to determine what alloy and what application technique would best achieve the desired low temperature bonding that is sought. Also additional tests are needed to determine the effects of moderate temperature and pressure increases.

The first attempt with copper foil was unsuccessful, but additional tests are needed to determine if this method might be satisfactory with other foils such as gold or silver, and with slightly elevated temperatures.

D. F. Fitzpatrick (Signature) 7-23-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
W. D. Johnston	11	S-15	S-74	672S91-01	700-22

DATE IN	DATE COMPLETED	PERFORMED BY
2-25-64	Interim	W. G. Grenier

NAME OF TEST

Photomicrographic Examination, Interim Report

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Article Examined:
Micro Electronics, Solid State Integrated Circuit.

EQUIPMENT INVOLVED:

Unitron Metallograph
B & L Research Metallograph
Photographic Dark Room facilities.

RESULTS:

See Figures 1-14 incl., Pgs. 3-11 incl. of the data

W. G. Grenier 3-27-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

PROCEDURE: GENERAL

Under the above Service Request No., a Micro Electronic, Solid State Integrated Circuit was examined Microscopically. As a result of this examination the Originator requested that the details of the circuit be presented in photographic form and Figures 1-3 incl. of the data were prepared for his perusal.

In his examination of Figures 2 and 3, the Requestor noted 13 specific anomalies. He therefore requested that those specific areas, encircled in Figures 2 and 3, be presented for his study at the highest practicable magnification and resolution. Figures 4-14 incl., of the data, present those areas at the magnification of 500 diameters. They were prepared using the B & L Research Metallograph and glass metallographic plates. Due to the configuration of the micro electronic circuit, as received, it was impossible to obtain a sufficiently flat field for higher magnifications.

W. G. Grenier 3-27-64

(Signature) *(Date)*

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

DATA: Composite Photomacrograph

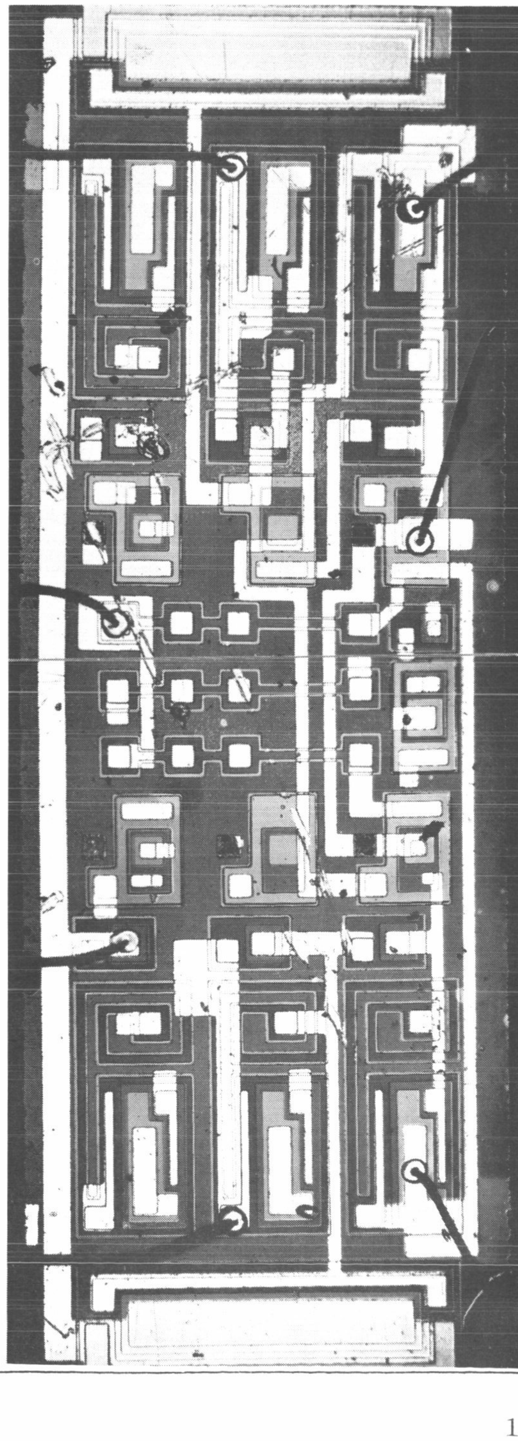


Figure 1–Micro Electronics, Solid State Integrated Circuit at 40 magnifications.

Photomacrograph from plate prepared on the Unitron Metallograph using the macroscopic lenses and accessories.

670-18 (2/64)

W. G. Grenier
(Signature)

3-27-64
(Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

DATA: Enlargement of Figure 1

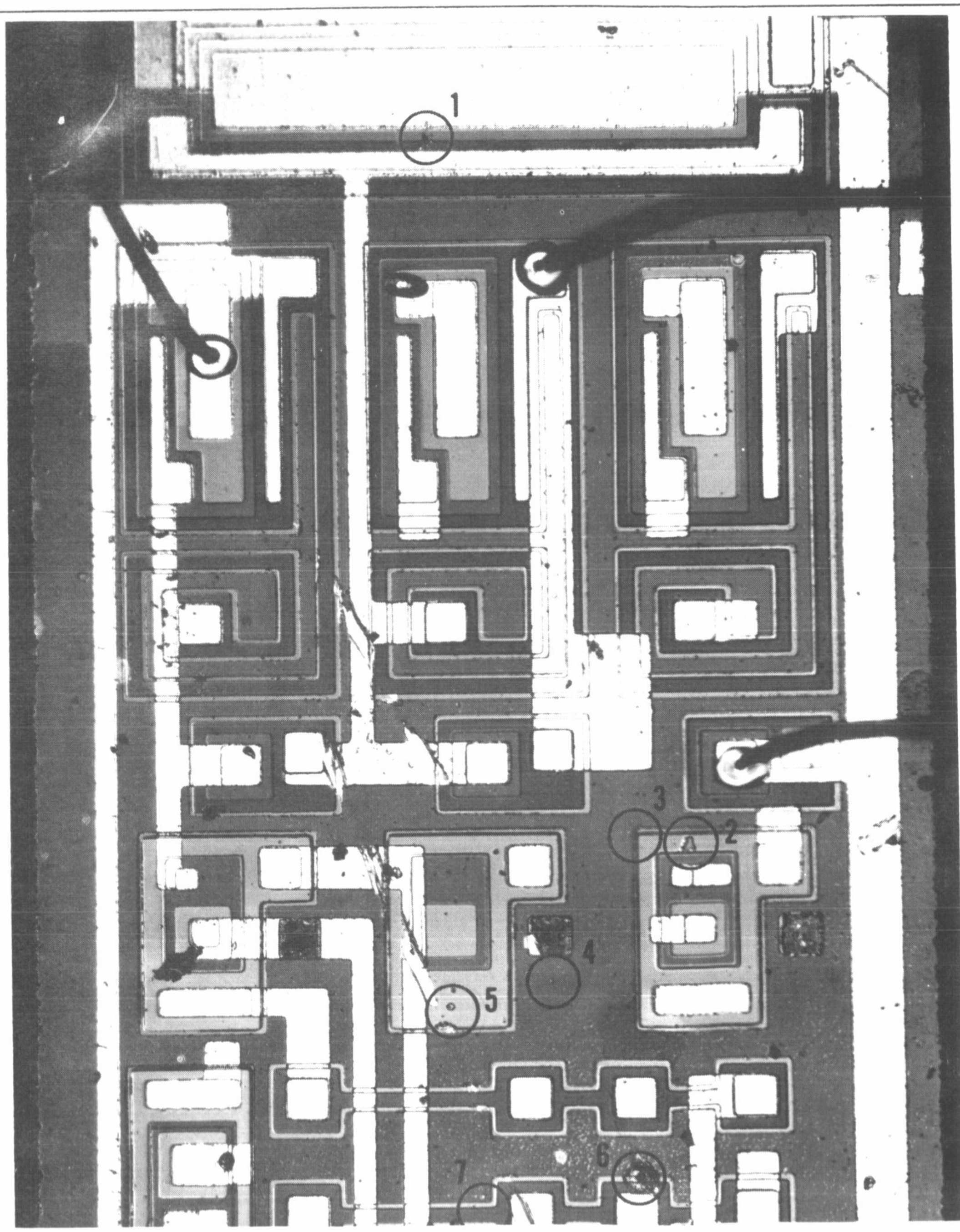


Figure 2-1/2 Figure 1, enlarged approximately by 2 diameters. The reference numbered circles, indicate locations which will be examined at much higher magnifications.

670-18 (2/64)

W. G. Grenier (Signature) 3-27-64 (Date)

NO. 1

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

DATA: Enlargement of Figure 1

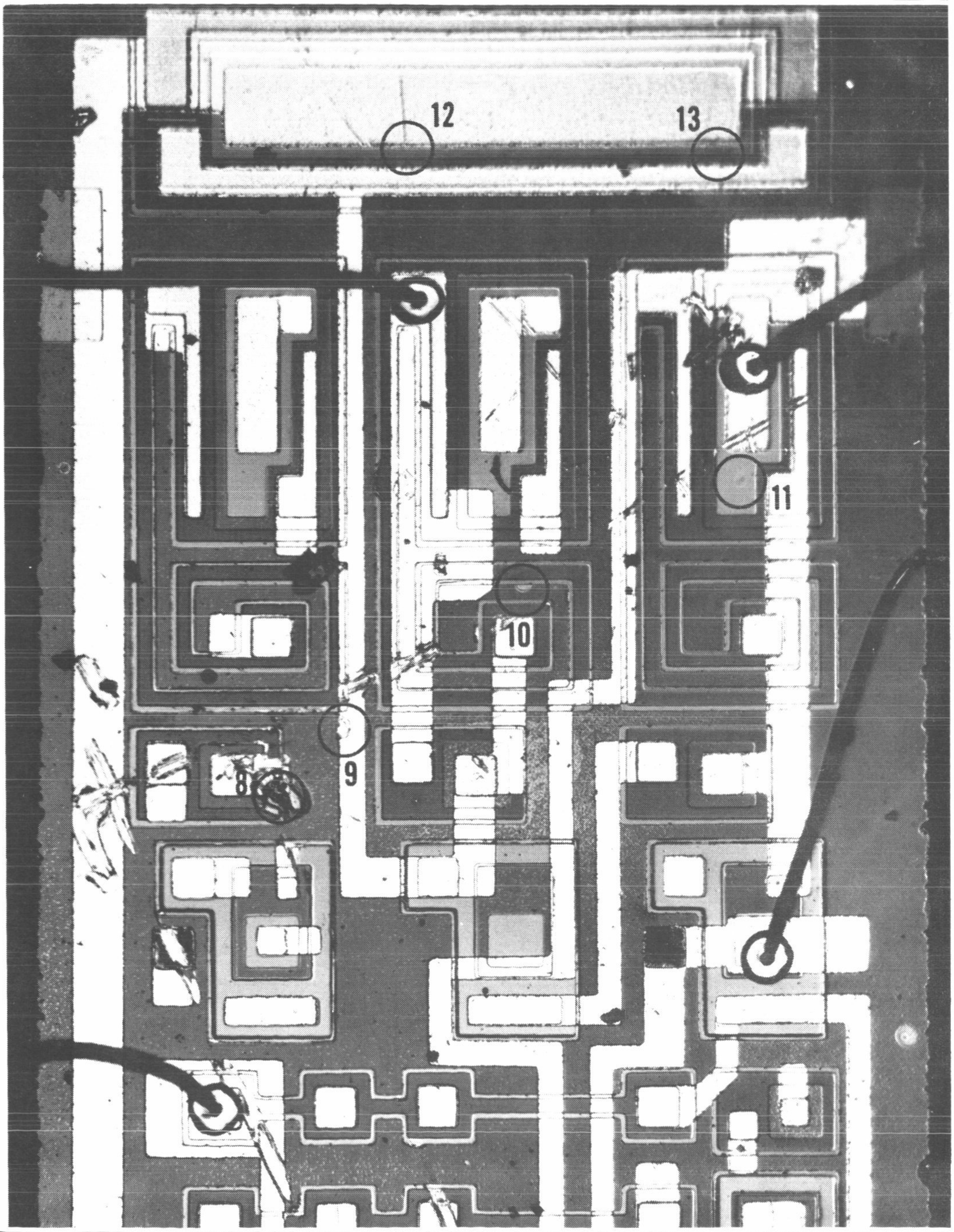


Figure 3—Other 1/2 of Figure 1, enlarged approximately 2 diameters. The reference numbered circles, indicate areas which will be examined at higher magnifications.

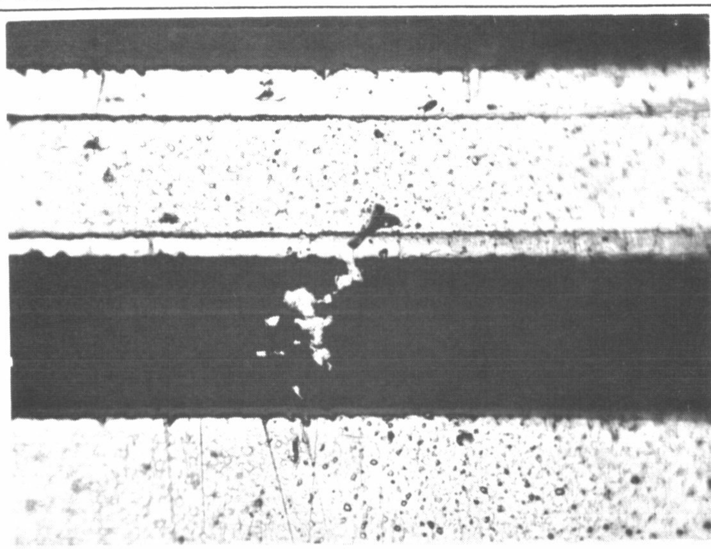
670-18 (2/64)

W. G. Grenier (Signature) 3-27-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

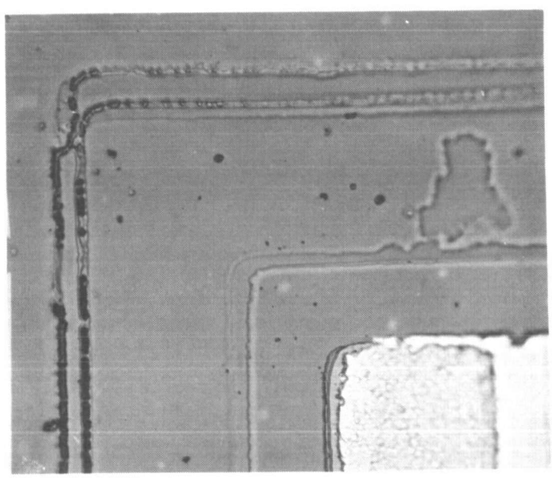
DATA: 500 Diameter Magnification Photomicrographs. Reference Encircled Area Numbers on Figures 2 and 3.



Area #1

Figure 3

Area #3



Orientation matches that of Figure 2.

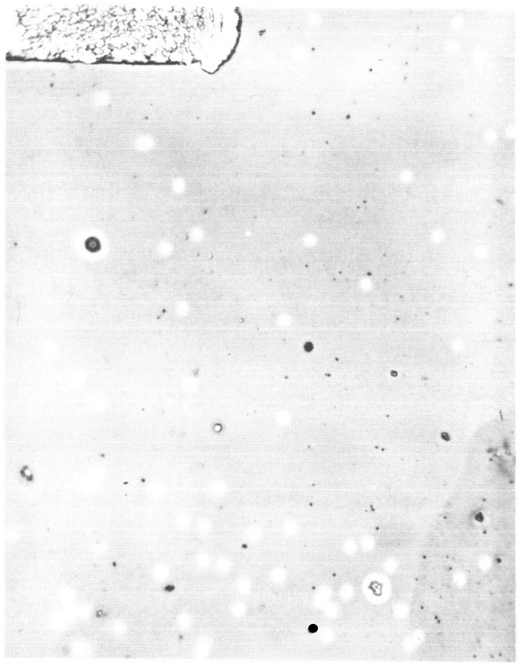
670-18 (2/64)

W. G. Grenier 3-27-64
(Signature) (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

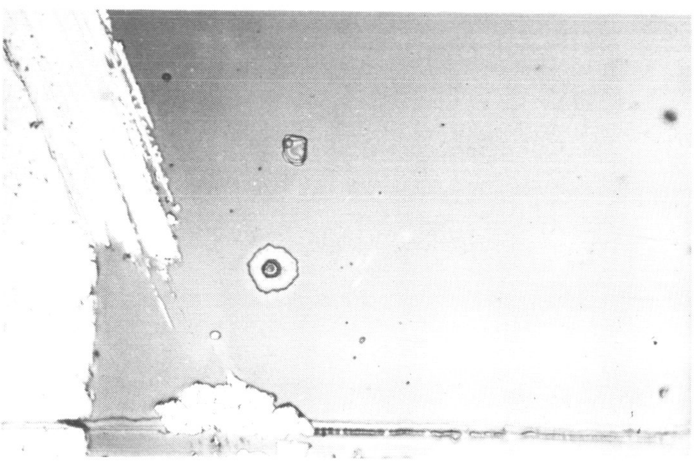
ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

DATA: 500 Diameter Magnification Photomicrographs. Reference Encircled Area Numbers on Figures 2 and 3.



Area #4

Figure 5



Area #5

Figure 6

Orientation matches that of Figure 2.

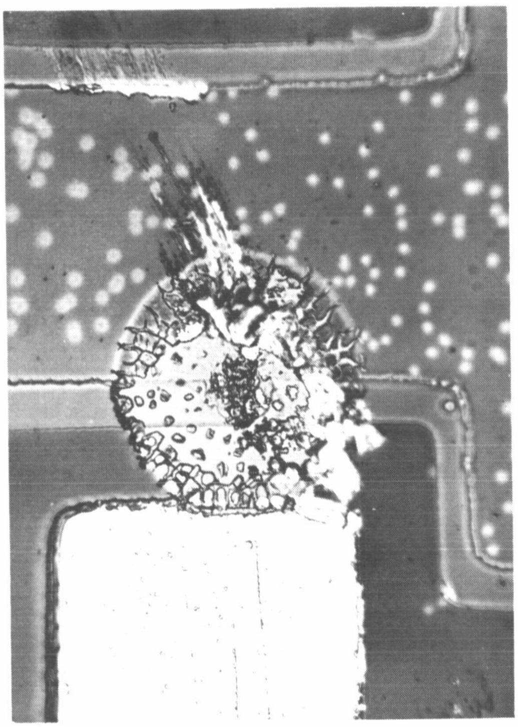
670-18 (2/64)

W. G. Grenier (Signature) 3-27-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

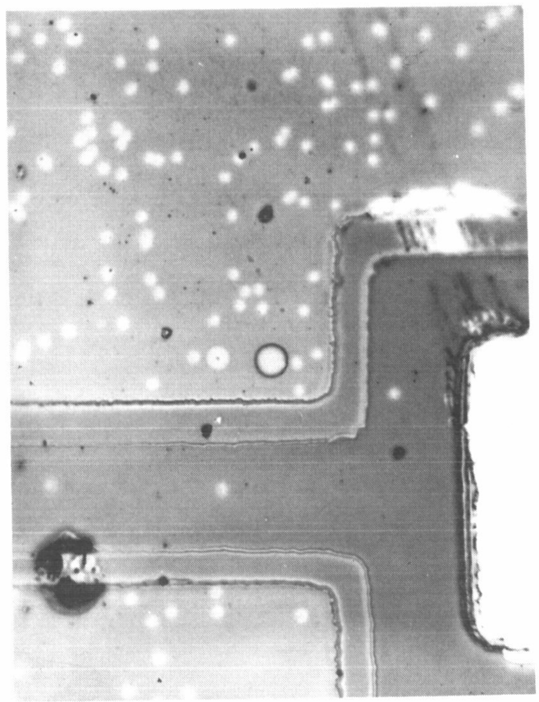
ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

DATA: 500 Diameter Magnification Photomicrographs. Reference Encircled Area Numbers on Figures 2 and 3.



Area #6

Figure 7



Area #7

Figure 8

Orientation matches that of Figure 2.

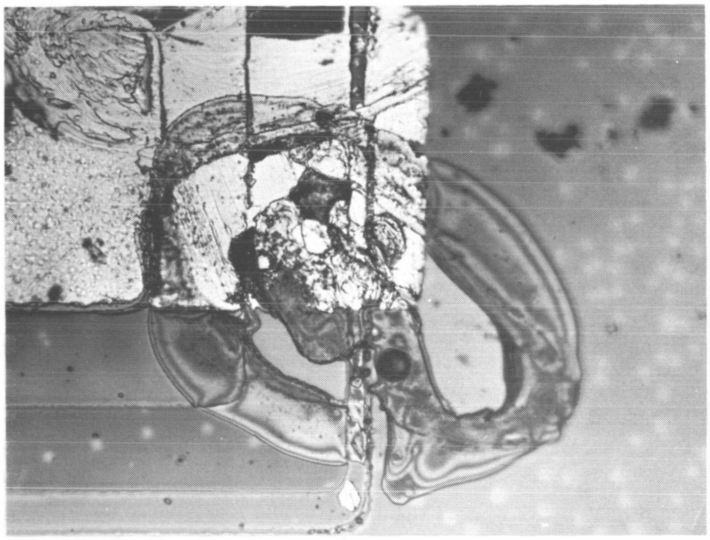
670-18 (2/64)

W. G. Grenier (Signature) 3-27-64 (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

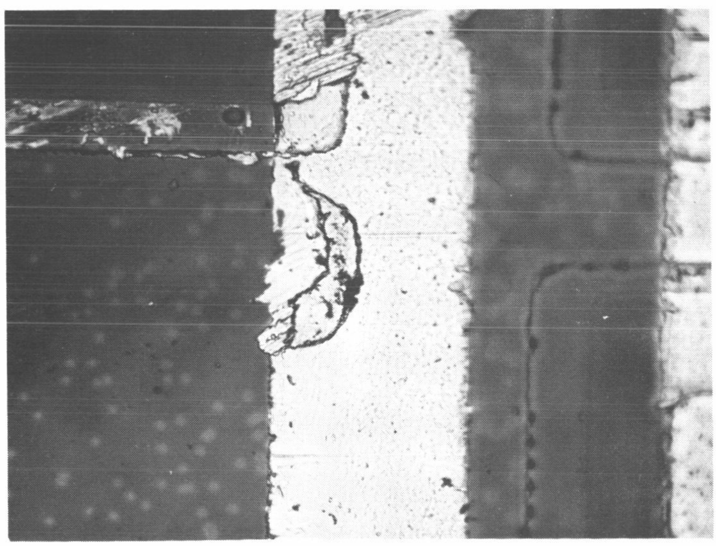
ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

DATA: 500 Diameter Magnification Photomicrographs. Reference Encircled Area Numbers on Figures 2 and 3.



Area #8

Figure 9



Area #9

Figure 10

Orientation matches that of Figure 3.

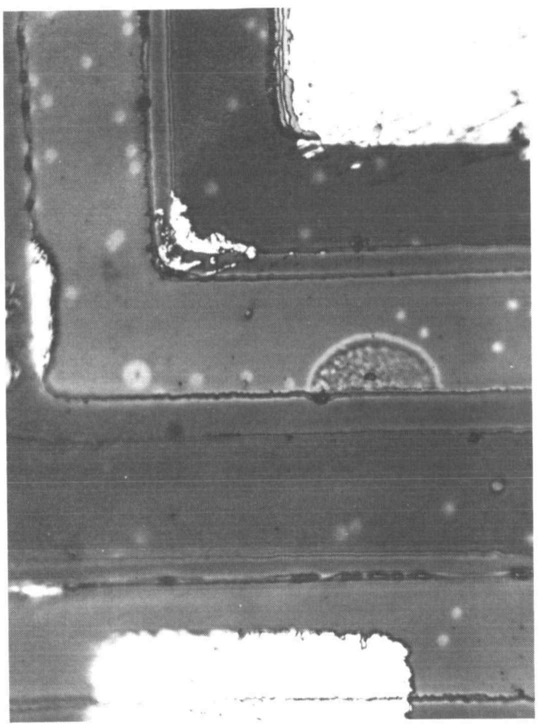
670-18 (2/64)

W. G. Grenier (Signature) 3-27-64 (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

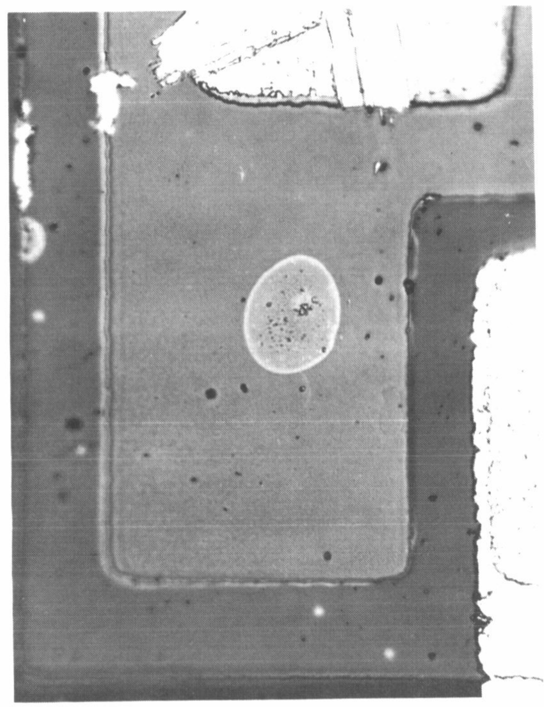
ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

DATA: 500 Diameter Magnification Photomicrographs. Reference Encircled Area Numbers on Figures 2 and 3.



Area #10

Figure 11



Area #11

Figure 12

Orientation matches that of Figure 3.

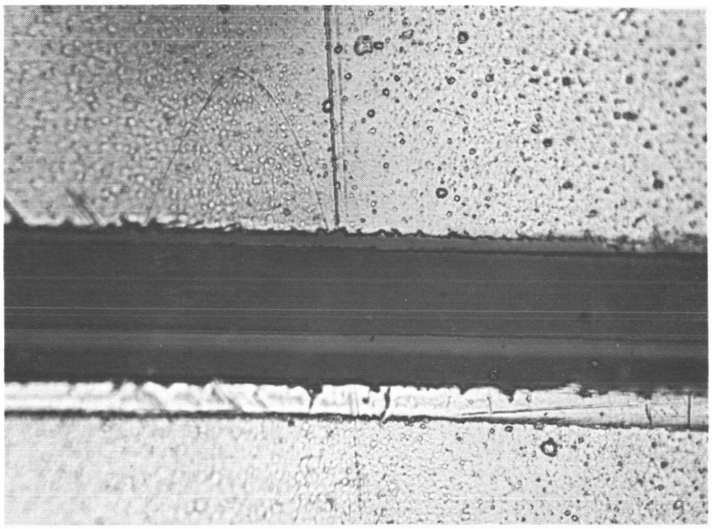
670-18 (2/64)

W. G. Grenier (Signature) 3-27-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

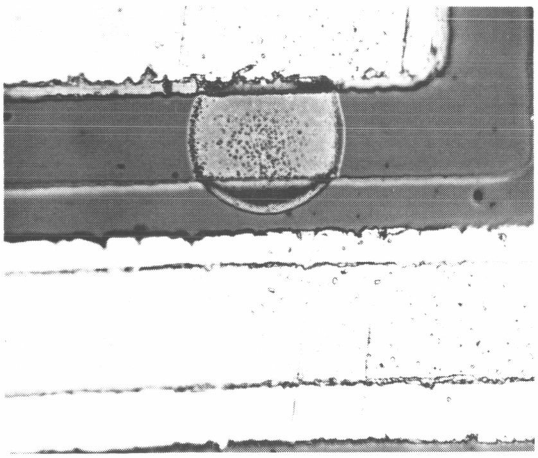
ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston	S-74	672S91-01	700-22

DATA: 500 Diameter Magnification Photomicrographs. Reference Encircled Area Numbers on Figures 2 and 3.



Area #12

Figure 13



Area #13

Figure 14

Orientation matches that of Figure 3.

670-18 (2/64)

(Signature) 3-27-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
W. D. Johnston, III	11	S-15	S-74	672S91-01	700-22

DATE IN	DATE COMPLETED	PERFORMED BY
4-7-64	4-16-64	W. G. Grenier

NAME OF TEST

Metallographic, Failure Study; Planar Diffused Transistors.

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Articles: 2 Silicon, planar diffused, transistors.

EQUIPMENT INVOLVED:

1. B & L Research Metallograph
2. Photographic Dark Room equipment.

RESULTS:

See Figures 1-4 inclusive.

W. G. Grenier (Signature) 4-13-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston, III	S-74	672S91-01	700-22

PROCEDURE: GENERAL

Two Micro Electronics, Silicon Diffused Transistors, were submitted by the Originator for microscopic examination. One transistor functioned while the other had failed. The failed transistor contained an internal short, not originally present, plus a loose connection at an external binding post.

It was desired that a photomicrograph of each transistor be prepared at magnifications of 100 diameters, or better, for the Originators perusal. It was impossible to obtain direct photomicrographs at magnifications exceeding 100 diameters. This was due to the particular configurations of the transistors, as mounted in their respective circuit boards. Therefore, photomicrographs at 100 diameters, were prepared on metallographic plates, using the B & L Research Metallograph. Contact prints were prepared using standard photographic dark room procedures and are presented in Figures 1–2 of the data. Enlargements were prepared of each photomicrograph, using the Omega D-2V enlarger and the metallographic plates. The enlargements are approximately 3 times the original magnifications and are presented as Figures 3–4 of the data.

W. G. Grenier (Signature) 4-13-64 (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston, III	S-74	672S91-01	700-22

DATA: Photomicrographs at a Magnification of 100 Diameters

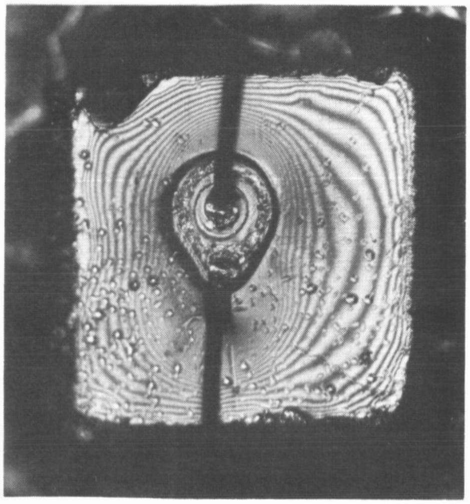


Figure 1

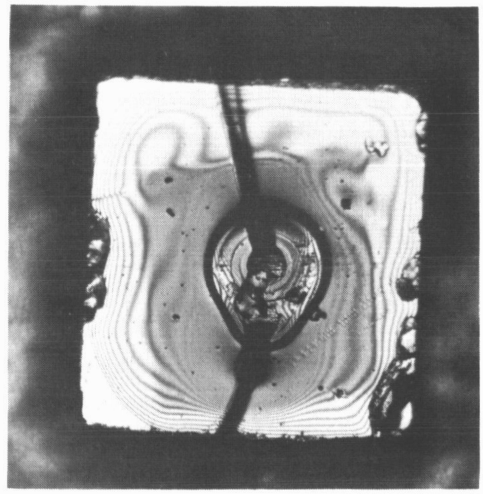


Figure 2

Silicon, Planar Diffused, Transistors at 100 magnifications.

Figure 1 shows that transistor which still functioned.

Figure 2 shows that transistor which had failed. Note apparent bridge.

670-18 (2/64)

W. G. Grenier (Signature) 4-13-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston, III	S-74	672S91-01	700-22

DATA: Enlargement of Photomicrograph in Figure 1.

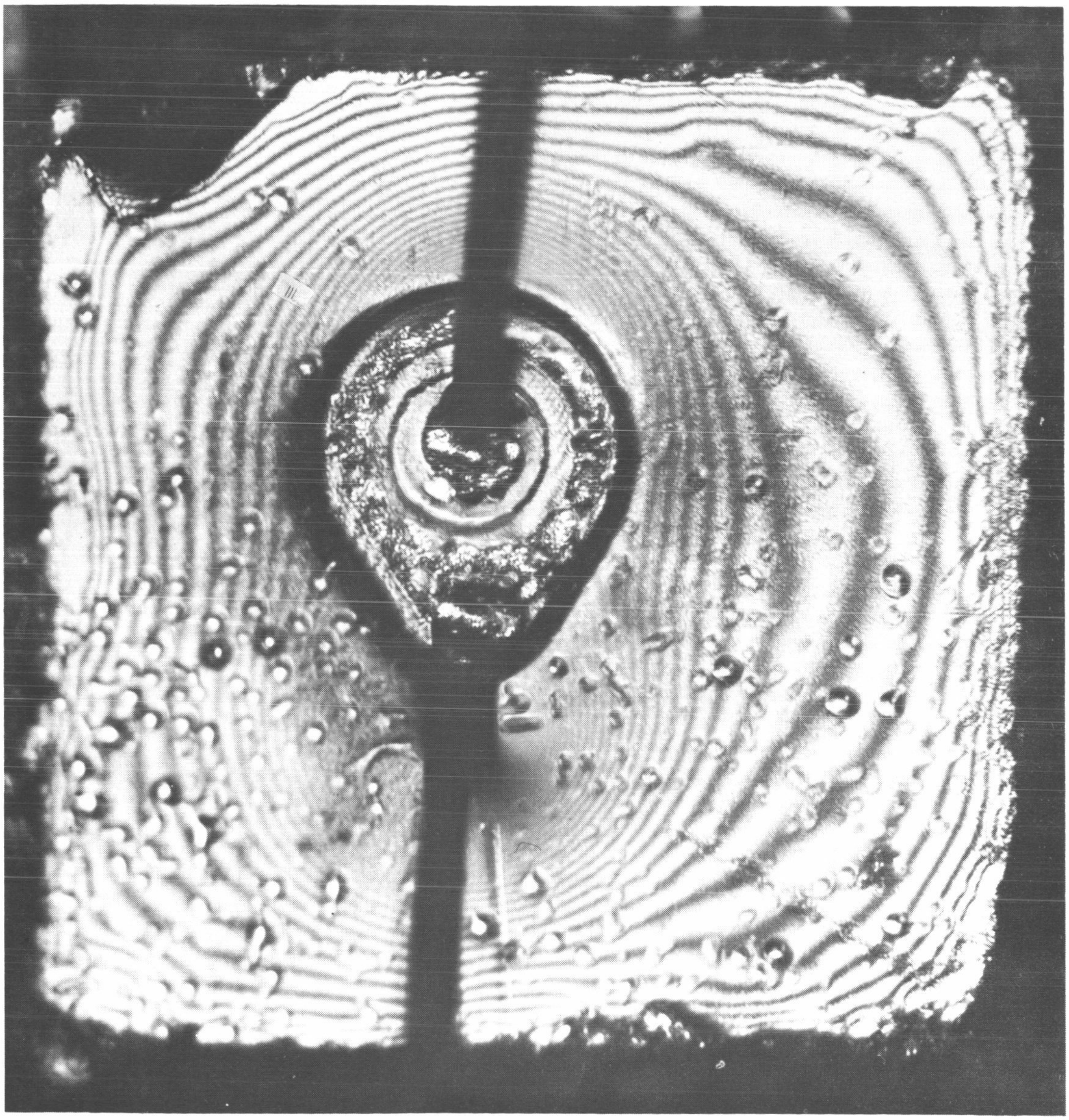


Figure 3—Good, Silicon, Planar Diffused Transistor Approximate Magnification = 300X.

670-18 (2/64)

W. G. Grenier (Signature) 4-13-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
W. D. Johnston, III	S-74	672S91-01	700-22

DATA: Enlargement of Photomicrograph in Figure 2.

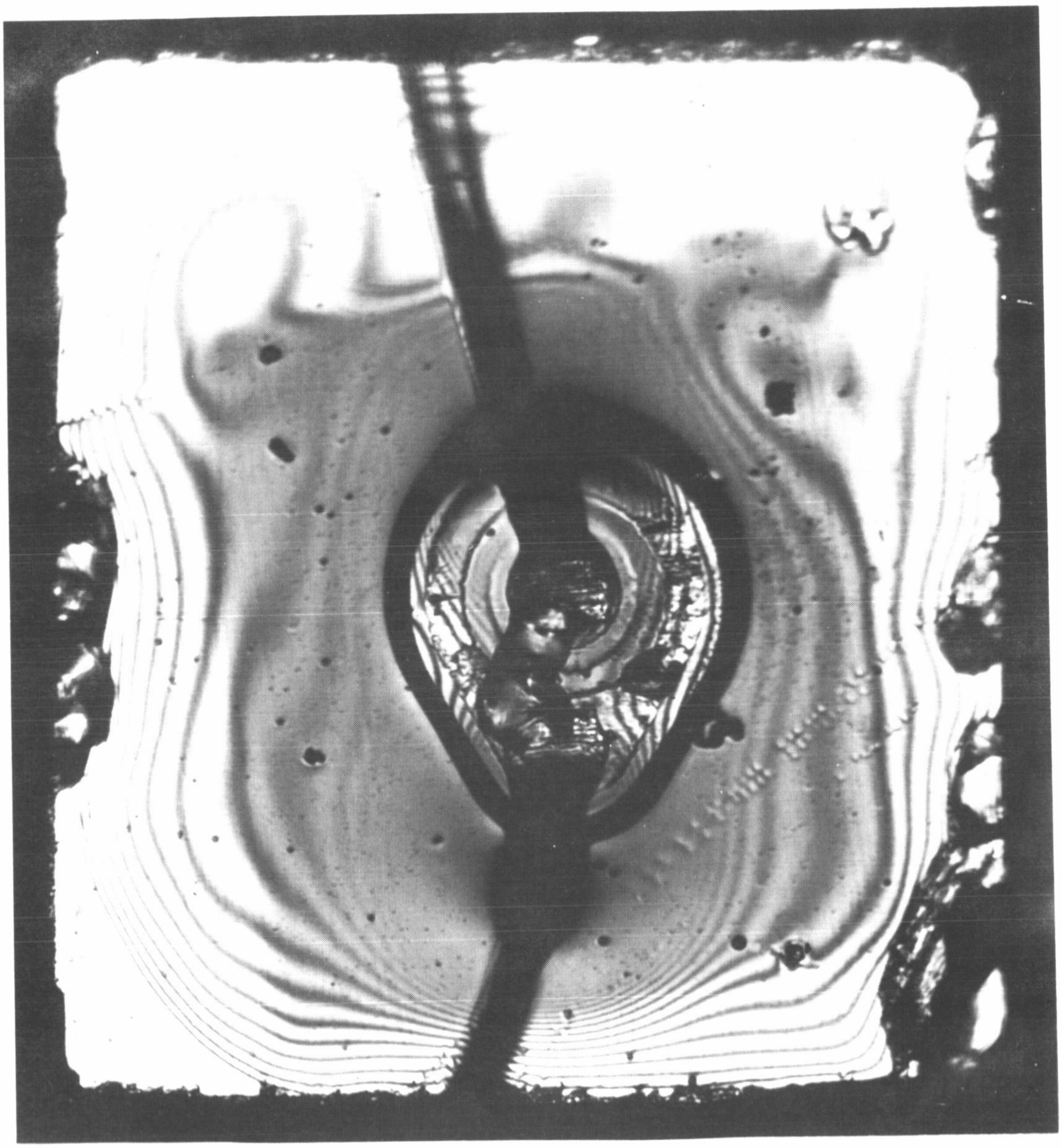


Figure 4–Failed – Silicon, Planar Diffused Transistor Approximate Magnification = 300X.

670-18 (2/64)

4-13-64

(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
C. E. Vest	11	S-120	OGO	673S70-01	810-5

DATE IN	DATE COMPLETED	PERFORMED BY
6-3-63	6-15-64	W. G. Grenier

NAME OF TEST

Tests of Special Coatings on Mg and Al

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Sample No.	Coating Description, 2 Samples Each = 18 Samples Total
1	Iridite #15, Mix A
2	Iridite #15, Mix C, Irilac 1000, 1-9 mix
3	Iridite #15, Mix A, Irilac 1000, 1-4 mix
4	Iridite #15, Mix A, Irilac 1000, 1-2 mix
5	Iridite #15, Mix C
6	Iridite #15, Mix C, Irilac 1000, 1-4 mix
7	Iridite #14, 3 oz/gal., 3 min.
8	Iridite #15, Mix C, Irilac 1000, 1-2 mix
9	Iridite #14, 3 oz/gal, 1 min.

EQUIPMENT INVOLVED:

Belt Surfacer, Buehler Handimet hand polisher, Ohaus Triple Beam Balance, Buehler hand operated hydraulic press, Kiethly Instruments Model 503 Milliohmmeter.

RESULTS:

1. Results of 24 hour corrosion tests indicated no breakdown of protective film.
2. Electrical Resistance Measurement data, see sheets 6-11 inclusive.
3. For Abrasion Resistance test methods and results see sheets 12-17 inclusive.

W. G. Grenier (Signature) 6-15-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

PROCEDURE: GENERAL – SAMPLING

Eighteen samples, as described on sheet 1 were submitted by the Originator for coating tests. The tests are to determine electrical and abrasion resistance. Each sample submitted was 3" wide by 6" long.

Each sample was sheared, by staff shop personnel, into specimens approximately 3" × 3". Two specimens for each coating condition was submitted to Mr. John Quill of Test and Evaluation Department for corrosion tests. In the shearing operation, each sample was sandwiched between heavy manila paper to minimize damage to coatings.

Following initial shearing, each remaining 1/2 sample was sheared into specimens approximately 1-1/2" × 1-1/2" square. Each small specimen was carefully deburred using the 240 grit paper with copious quantities of water, on the Handimet hand polisher. Each small specimen was subsequently wrapped in tissue and placed in an envelope bearing the same number as the original sample.

It was initially assumed that the Irilac coating would have a very high electrical resistance. Therefore the Model 6105 Resistivity Adapter, was set up in conjunction with its associated Electrometer and a power supply. It was learned that in every case the electrical resistance of each coating was very small. Therefore a Keithley Model 503 Milliohmeter will be used to measure the electrical resistance of coatings and substrate

W. G. Grenier (Signature) 6-10-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

PROCEDURE: GENERAL – ELECTRICAL RESISTANCE MEASUREMENTS

materials. The specimens were placed between two solid cylinders of brass 3/4" in diameter. The system was placed in an hydraulic press fitted with a guage to read that pressure on the piston. Sufficient pressure is used to move the pressure indicating needle slightly, but not enough to read. The pressure used should be as nearly identical as possible so a zero mark will be used. From past experimentation results it is estimated that the compressive forces on each specimen will be on the order of 100 p.s.i., in the area of contact.

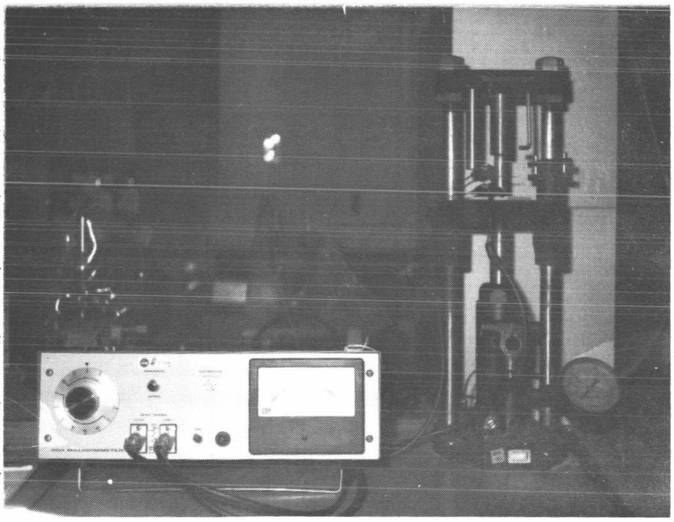


Figure 1

Typical test setup for electrical resistance measurements. Shows Model 503 Milliohmeter and hydraulic press with specimen in place.

W. G. Grenier (Signature) 6-10-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S73-01	810-5

PROCEDURE: GENERAL – ELECTRICAL RESISTANCE MEASUREMENTS (Continued)

Experimentation shows that some uniform amount of pressure must be applied, to brass cylinder-specimen system, to achieve any degree of uniformity. Excessive pressure appears to be equally deleterious, resulting in low resistance value.

670–15

W. G. Grenier 6-10-64
(Signature) (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. West	OGO	673S73-01	810-5

DATA: Conditions for Electrical Resistance Measurements

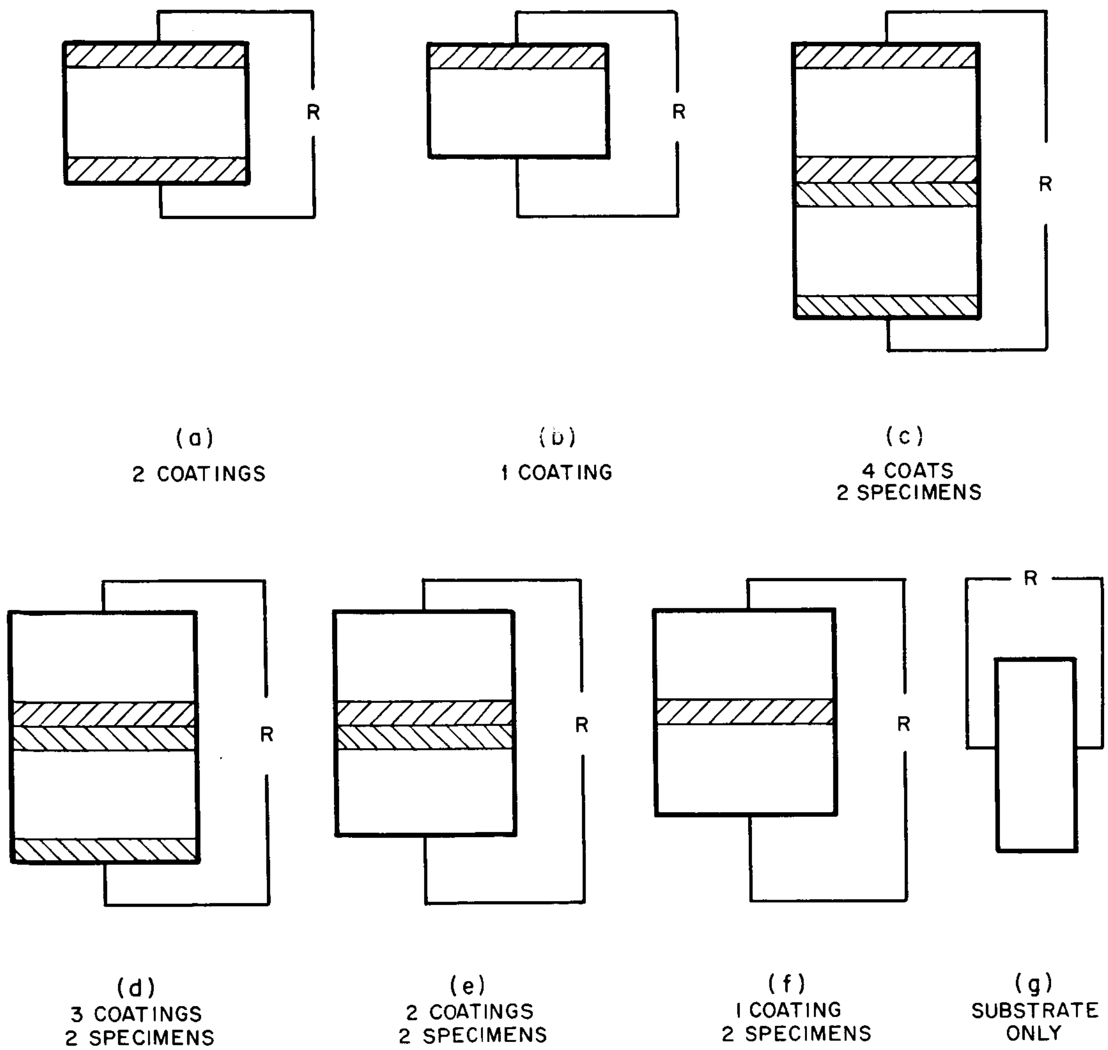


Figure 2

Schematic Diagrams of the Various Test Arrangements

670-18 (2/64)

W. G. Grenier (Signature) 6-10-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S73-01	810-5

DATA: ELECTRICAL RESISTANCE MEASUREMENTS

SHEET-5 CONDITION	RESISTANCE IN MILLIOHMS — READING NUMBER 1	2	3	4	5	SPREAD LOW-HIGH	AVERAGE		
			SPECIMEN NO. 1						
a	25	14.5	6	6.8	16.5	6 - 25			
b	15	18	14	16.5	12	12 - 18			
c	1600	850	910	360	970	360 - 1600			
d	630	560	460	470	550	460 - 630			
e	420	465	270	420	510	270 - 510			
f	56	76	125	220	95	56 - 220			
		Substrate, One (1) Sheet, No Coating							
g	0.54	0.56	2.2	2.9	5.6	0.54 - 5.6			
			SPECIMEN NO. 2						
a		6.9	5.6	7.6	9.6	5.6 - 9.6			
b	5.4	8.9	6.5	6.0	5.9	5.4 - 8.9			
c	165	125	150	100	125	100 - 165			
d	100	105	125	115	160	100 - 160			
e	185	92	110	130	170	92 - 185			
f	24	46	39	37	30	24 - 46			
g	4.7	2.3	6.1	7.2	5.9	2.3 - 7.2			

670-19 (2/64)

W. G. Grenier (Signature) 6-11-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

DATA: ELECTRICAL RESISTANCE MEASUREMENTS

SHEET-5 CONDITION	RESISTANCE IN MILLIOHMS — READING NUMBER 1	2	3	4	5	SPREAD LOW	SPREAD HIGH	AVERAGE		
	SPECIMEN NO. 3									
a	68	81	94	60	52	52	94			
b	37	27	36	40	28	27	40			
c	470	400	600	920	210	210	920			
d	440	320	280	420	395	280	440			
e	510	490	720	340	540	340	720			
f	92	98	115	115	90	90	115			
g	5.6	14	4.7	6.6	10.5	4.7	14			
	SPECIMEN NO. 4									
a	92	83	135	130	88	83	135			
b	72	68	85	89	110	68	110			
c	1200	1750	1150	750	1050	750	1750			
d	1050	1500	1100	920	1400	920	1500			
e	1250	1050	1300	1100	1000	1000	1300			
f	220	185	300	380	395	185	395			
g	2.4	4.7	1.7	2.5	5.9	1.7	5.9			

670-19 (2/64)

W. G. Grenier (Signature) 6-11-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

DATA: ELECTRICAL RESISTANCE MEASUREMENTS

SHEET-5 CONDITION	RESISTANCE IN MILLIOHMS — READING NUMBER 1	2	3	4	5	SPREAD LOW	SPREAD HIGH	AVERAGE		
SPECIMEN NO. 5										
a	15	17.5	17.5	15.5	17.5	15	17.5			
b	8.7	10.5	8.2	9.8	13.2	8.2	13.2			
c	100	115	110	100	110	100	115			
d	180	137	165	267	127	137	267			
e	130	177	122	120	120	120	177			
f	45	83	82	89	73	45	89			
g	2.95	3.7	3.9	2.9	3.7	2.9	3.9			
SPECIMEN NO. 6										
a	39	54	48	37	44	37	54			
b	9.4	15	8.3	8.2	8.9	8.2	15			
c	140	155	120	220	150	120	220			
d	145	149	120	130	110	110	149			
e	130	149	130	225	125	125	225			
f	52	59	59	60	67	52	67			
g	4.5	3.8	8.1	5.8	6.7	3.8	8.1			

670-19 (2/64)

W. G. Grenier (*Signature*) 6-11-64 (*Date*)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

DATA: ELECTRICAL RESISTANCE MEASUREMENTS

SHEET-5 CONDITION	RESISTANCE IN MILLIOHMS									
	READING NUMBER					SPREAD LOW-HIGH		AVERAGE		
	1	2	3	4	5					
SPECIMEN NO. 7										
a	143	320	165	100	265	143	320			
b	275	265	160	290	320	160	320			
c	1,750	6,800	6,200	10,000	2,800	1750	10,000			
d	32,000	46,000	21,000	4,200	6,000	–	–			
e	4,000	38,000	2,700	36,000	6,100	–	–			
f	31	62	18.5	100	54	18.5	100			
g	0.66	0.78	0.84	0.75	0.91	0.66	0.91			
SPECIMEN NO. 8										
a	35	53	52	74	52	35	74			
b	6.7	10.6	13.6	10.5	17	6.7	17			
c	420	470	380	270	610	270	610			
d	300	350	270	300	390	270	390			
e	230	350	170	310	275	170	350			
f	40	39	50	38	92	38	92			
g	5.7	3.5	3.2	7.6	7.4	3.2	7.6			

670-19 (2/64)

W. G. Grenier (Signature) 6-11-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-5	810-5

DATA: ELECTRICAL RESISTANCE MEASUREMENTS

SHEET-5 CONDITION	RESISTANCE IN MILLIOHMS READING NUMBER 1	2	3	4	5	SPREAD LOW-	HIGH	AVERAGE
SPECIMEN NO. 9								
a	123.5	125	110	160	105	105	160	
b	17.4	16.5	31	15.5	13.5	13.5	31	
c	285	7900	365	220	7200	220	7900	
d	1850	690	8400	2150	1900	690	8400	
e	510	275	380	610	420	275	610	
f	55	110	44	46	360	44	360	
g	12.5	11	19.5	13	15	11	19.5	

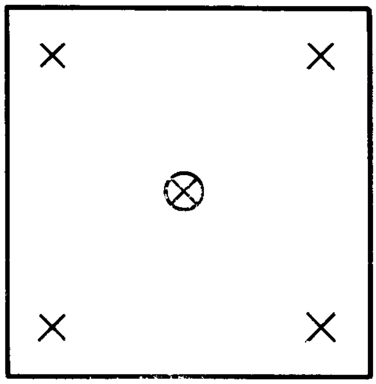
670-19 (2/64)

W. G. Grenier (*Signature*) 6-12-64 (*Date*)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

DATA: Electrical Resistance, Reading Number Locations



TYPICAL SPECIMEN 1 1/2" x 1 1/2"

X READING NUMBERS 1-4 INCL IN EVERY CASE

⊗ LOCATION OF READING NO. 5, IN EVERY CASE

Figure 3

670-18 (2/64)

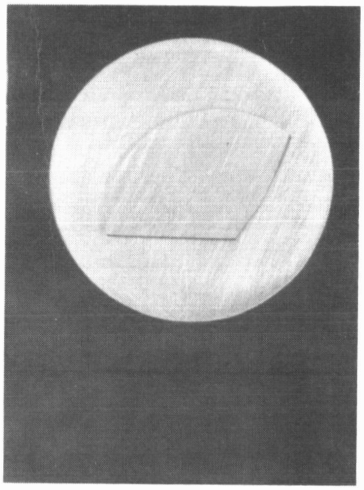
W. G. Grenier (Signature) 6-12-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

PROCEDURE:



← 28 gm weight fastened to movable section coated on both sides.

← Specimen being tested, with coating removed on underside.

Figure 4–Abrasion Test Method No. 1

Initial testing arrangement for abrasion tests. The top portion, weighted, was moved laterally over the test specimen. As the specimens were only 1-1/2 inches square, the stroke was very short. No accurate determination of stroke length was possible due to the fact that this was a hand operation.

This method was discontinued, due to the erratic results, as shown in the data on sheet 13 of this report.

W. G. Grenier 6-12-64
(Signature) (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

DATA:

ABRASION TESTS
RESULTS OF METHOD NO. 1, SPECIMEN #1

CYCLE NO. OF	RESISTANCE MILLIOHMS	REMARKS
0	26.6	———
10	29.5	Some abrading evidenced on specimen edges.
30	43	Edges show marked indications of abrasion
50	34	———
100	130	———
150	100	Severe abrasion of edge areas apparent to the eye.
200	140	———
300	155	Discontinue

Electrical resistance was measured in the center only of the specimen utilizing the same arrangement as for the Electrical Resistance measurements.

Cycle = One stroke forward and one stroke back.

670-19 (2/64)

W. G. Grenier (Signature) 6-12-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

PROCEDURE: ABRASION TESTS, METHOD NO. 2

This method will employ the same principle as method No. 1, in that the coating will be abraded against another coating, of the same type, under a constant load of 28 grams. A sample of material from the Corrosion Tests will be used as the base. The specimen to be tested will be polished through the 600 grit paper on one surface, to remove that coating and present one clean side. The 28 gram weight will be affixed to the clean side of the test specimen. The remaining coated surface will be placed in contact with the coated surface of the 3" × 3" sample. The test specimen will be caused to move backward and forward, with no weight other than the 28 grams. The specimen will be removed from the weight at periodic intervals, cleaned by an air blast and the electrical resistance in the geometric center measured. This permits specimen travel of approximately 5" per cycle.

W. G. Grenier (Signature) 6-12-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

DATA: ABRASION TEST, RESULTS OF TEST METHOD NO. 2
SPECIMEN #1 ON SAMPLE #1

CYCLES NO. OF	RESISTANCE MILLIOHMS	REMARKS
0	51	——
10	56	——
20	41	——
50	32	Edges are abraded through coating
100	29	——
200	31	——
300	44	——
400	30	Area of coating removed, away from center
450	29	——
500	27	——
600	16	6 in spot off center
700	8	——
800	8	——
1000	4.9	——
1200	6.2	——
1500	7.3	——

Testing stopped - Coating was completely removed by abrasive papers and the Electrical Resistance checked in the same test arrangement as used throughout.

Resistence of the uncoated and cleaned substrate = 0.51 milliohms.

670-19 (2/64)

W. G. Grenier (Signature) 6-12-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

DATA: ABRASION TEST, RESULTS OF TEST METHOD NO. 2
SPECIMEN #2, ON SAMPLE NO. 2

CYCLES NO. OF	RESISTANCE MILLIOHMS	REMARKS
0	8.8	
50	15.5	Small areas in corners worn to substrate
100	20.5	Edges wearing—corner areas enlarging
200	23.5	———
300	30	———

Discussed with Originator. He recommended that electrical resistance measurements be discontinued and visual checks be used instead. Therefore all subsequent tests will be based on number of cycles to failure. Failure will be considered as having occurred when substrate can be clearly defined by visual observation. Therefore no coating will be removed prior to testing. One and one half inch square specimen will still be abraded in manner described in Test Method No. 2, sheet 14.

670-19 (2/64)

W. G. Grenier (Signature) 6-15-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

DATA: ABRASION TEST RESULTS, VISUAL CHECK

TEST NO.	SPECIMEN NO.		CYCLES TO FAILURE			TEST NO.	SPECIMEN NO.		CYCLES TO FAILURE	
1	1		2			13	7		12	
2	1		5			14	7		10	
3	2		5			15	8		3	
4	2		3			16	8		3	
5	3		4			17	9		5	
6	3		9			18	9		6	
7	4		5							
8	4		3							
9	5		5							
10	5		1							
11	6		5							
12	6		5							

Note: See sheet 16 for definition of failure.

670-19 (2/64)

W. G. Grenier (*Signature*) 6-15-64 (*Date*)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-5

CONCLUSIONS

The nine coatings listed on page 1 were tested for corrosion resistance, electrical resistance and abrasion–scratch resistance. Substrate materials were Mg (Nos. 1, 2, 3, 4, 5, 6 and 8) and Al (Nos. 7 and 9).

Specimens subjected to the OGO Humidity test (93% min. R.H., 40°C and 24 hrs) showed no corrosion product. Specimens subjected to electrical resistivity test showed increasing resistivity with the coating with Iridite + Irilac with increasing lacquer content (Iridite in a lacquer coating). Specimens subjected to abrasion-scratch test showed no significant difference in abrasion-scratch resistance. Coating are not very abrasion-scratch resistant.

The results of the electrical and the abrasion-scratch resistance tests were for comparative purposes only. Further testing of these coatings is advisable before approval or acceptance for space use can be stated.

C. E. Vest — 6-15-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
C. E. Vest	11	S-120	OGO	673S70-01	810-8

DATE IN	DATE COMPLETED	PERFORMED BY
7-16-64	7-29064	W.G. Grenier, and D. Freedman

NAME OF TEST

Niobium to 1100 Aluminum, Ultrasonic Weld Examination.

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Metallographic preparation and photomicrographic presentation of 0".005 thick Niobium strip welded to aluminum (1100) sheet approximately 0".020 thick.

Two specimens identical except for:

Spec. #1 – Al was Nickel flashed prior to weldment
Spec. #2 – No nickel flash was employed on the Al.

EQUIPMENT INVOLVED:

B & L Model L Macro Camera with Polaroid Back, Abrasive cutoff Wheel, Glass filled epoxy mounting medium Wet belt surfacer, Handimet Hand polisher, Buehler lapping oil, Two wheel hand polishing table, 600 grit Aluminum Oxide abrasive, Magomet magnesium oxide abrasive, Hydrofluoric acid, Ammonium Fluoride, Bausch & Lamb Research Metallograph, Metallographic plates, well equipped darkroom facility.

RESULTS:

1. Photomacrographs at 1-1/2×, see sheets numbers 3 and 4.
2. Photomicrographs at 500×, see sheets numbers 8, 9, and 11, which show no weld fusion zone.

W. G. Grenier — 7-29-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

PROCEDURE: GENERAL

I. PHOTOMACROGRAPHS

Photomacrographs, at a magnification of approximately one and one-half diameters, were prepared of Samples Numbered 1 and 2, in the as received condition. The Bausch & Lamb, Model-L camera, equipped with a 158 mm lens and Polaroid-500 camera back was used for both samples. The resultant macrographs are presented in Figures 1–4 inclusive on sheets numbered 3 and 4 of this report.

II. SECTIONING OF SAMPLES

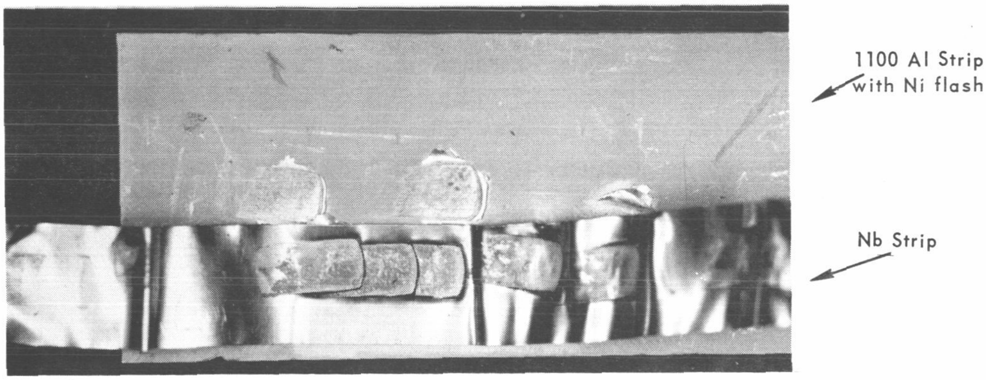
The samples were sectioned as per instructions under item 3, of the Originators additional information sheet. The abrasive cutoff wheel was used, with maximum coolant flow, and with as slow a feed as was practicable with the hand feed available. Due to the minute size of the actual specimens areas involved, it was not possible to obtain all the sections desired by the Originator. From Sample No. 1, two longitudinal and one transverse, sections were obtained. From Sample No. 2, on longitudinal and two transverse sections were obtained. In the instance of Sample No. 2; upon completion of the longitudinal cut, it was learned that the Niobium strip had peeled off of the aluminum base metal. This occurred in spite of a very slow hand feed.

W. G. Grenier (Signature) 7-20-64 (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

DATA: Photomacrographs; Sample No. 1



1-1/2X

Figure 1—Front Surface, Showing Apparent Welded Area.

1-1/2X

Figure 2—Back Surface, Showing Apparent Weld Area Penetration or Pressure Points.

Sample No. 1 — As Received.

W. G. Grenier (Signature) 7-20-64 (Date)

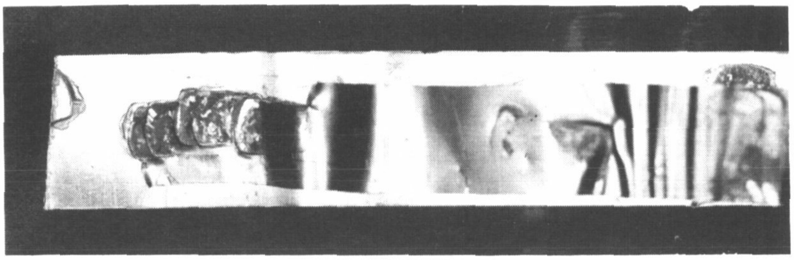
NO. I

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

DATA: Photomacrographs; Sample No. 2

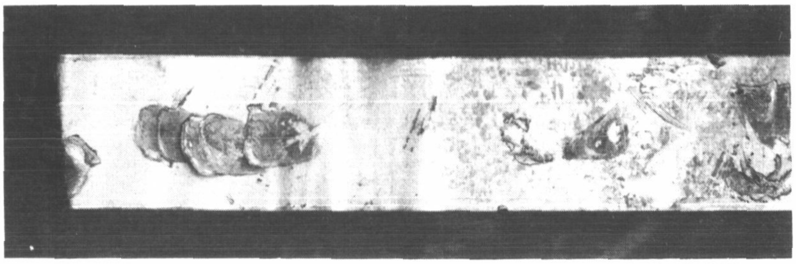


1100 Al strip with no Ni flash

Nb Strip

1-1/2X

Figure 3–Front Surface Showing Welded Area At One End.



1-1/2X

Figure 4–Backside of Figure No. 3.

Sample No. 2, As Received

670-18 (2/64)

W. G. Grenier (Signature) 7-20-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

PROCEDURE: GENERAL

III. MOUNTING FOR METALLOGRAPHY

Due to the thinness and irregularities of the specimens, they will be mounted in a glass filled epoxy mounting medium. To prepare the glass filled epoxy, blend 3 parts, by weight, of Hysol-2038 epoxy with 2 parts, by weight, of 325 mesh ground glass. Place the epoxy and ground glass blend in a sealed container and permit to stand for a minimum time of 24 hours. This action is to relieve trapped air. After the 24 hour waiting period, mix Hysol-3404 hardener with the foregoing mix in the ratio of one part hardener to ten parts mix, by weight. Blend thoroughly.

Place the specimens in bakelite ring forms. Pour the epoxy mixture, carefully over and around each specimen. Each specimen will be so located in the ring form that the face to be observed will be at the bottom. The epoxy mixture will be poured into the ring form until said form is slightly overfilled. Slight overfilling is accomplished when the epoxy mixture forms a dome shaped appearance above the edges of the ring form. Permit to cure 24 hours, before grinding.

When the sealed container of epoxy and glass was opened, it was observed that the surface appeared frothy with air bubbles. Upon pouring a portion of the mix into a paper cup, prepatory to adding hardener, it was observed that bubbling continued after a lapsed

W. G. Grenier (Signature) 7-22-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

PROCEDURE: GENERAL

III. MOUNTING (Continued)

time of 5 minutes. It is to be observed that difficulty in obtaining fully hardened specimen mounts has occurred in the past, when using this medium.

Due to the apparent irreplacability and small quantity of specimen material available, it is obvious that the first attempt at mounting must be adequate. Therefore, the blended glass and epoxy base will stand until no air bubbles are apparent on its surface.

Twenty four hours later (0900, 7-22-64) no air bubbles were apparent, so specimens were prepared as per Sheet 5.

IV. POLISHING

Having permitted the glass and epoxy mounts to harden and cure at room temperature for 48 hours, they were sufficiently hard, to be considered good mount.

Polishing of the specimens was conducted as for straight aluminum. This was as directed by the Originator. Rough grinding was accomplished using the wet belt surfacer,

W. G. Grenier (Signature) 7-27-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

PROCEDURE: GENERAL

IV. POLISHING (Continued)

with an 80 grit Silicon carbide belt and copious quantities of water. Find grinding was done on the Handimet hand polisher. New silicon carbide papers were installed and Buehler Diamond Lubricant was used in lieu of Kerosene.

Fine polishing was accomplished using the two wheel polishing table. The first wheel was a conventional bronze wheel, using billiard cloth charged with 600 grit aluminum oxide, in a soapy water extender. Final polishing was done on the low speed wheel. For this an aluminum wheel covered with Micro cloth was used in conjunction with Magomet abrasive, soap, and distilled water. Following this step the Originator studied each specimen and photomicrographs of each specimen were prepared as directed. Those photomicrographs, taken in the as-polished condition, at a magnification of 500 diameters, are presented as Figures 5-8 inclusive, on sheets 10 and 11. The Niobium surface is typical for the polishing procedures employed. Polishing behaviour indicated that there was no alloying effect of Nb-Al or Nb-Ni.

W. G. Grenier 7-27-64
(Signature) (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

DATA: Photomicrographs, Sample No. 1, as polished at 500×, Weld cross sections

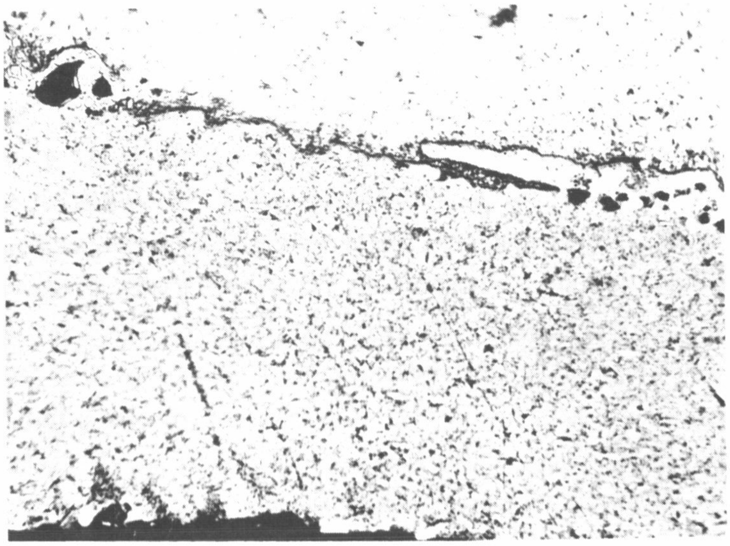


Figure 5–Sample No. 1, Longitudinal Section

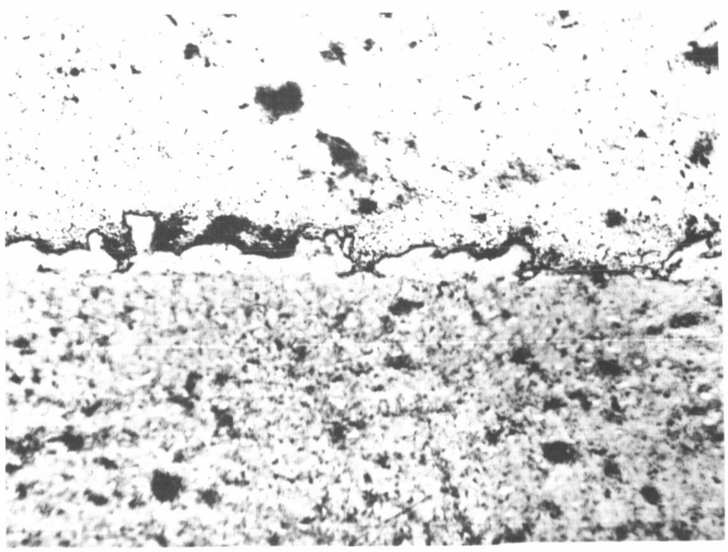


Figure 6–Sample No. 1, Transverse Section

670-18 (2/64)

W. G. Grenier (Signature) 7-29-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

DATA: Photomicrographs, Sample No. 2, as polished at 500×, weld cross sections

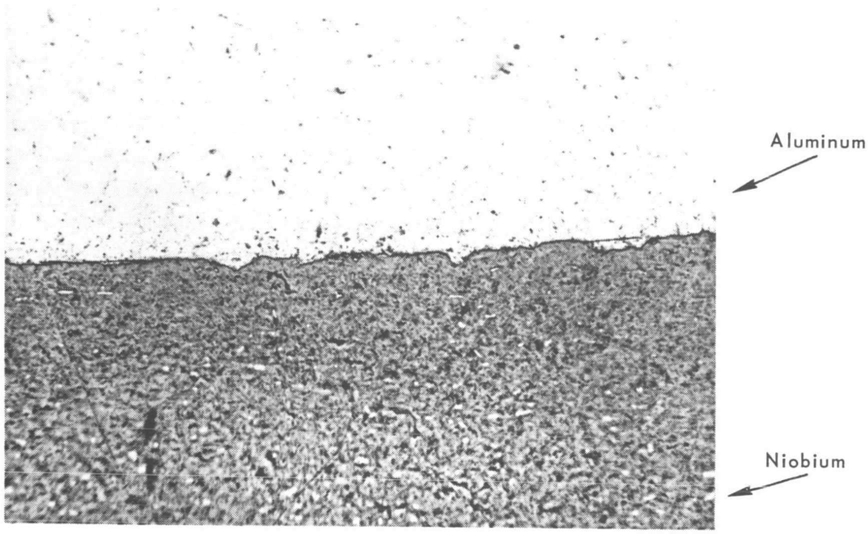


Figure 7–Sample No. 2, Longitudinal Section

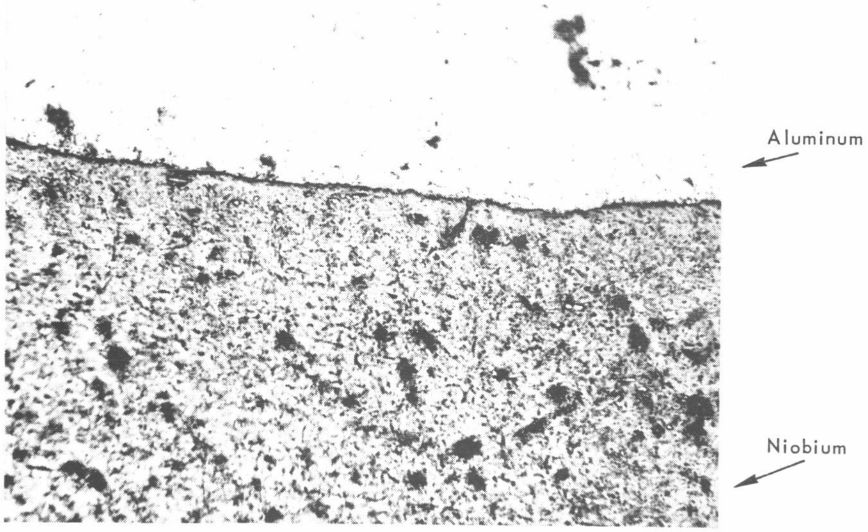


Figure 8–Sample No. 2, Transverse Section

670-18 (2/64)

W. G. Grenier (Signature) 7-29-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

PROCEDURE: METALLOGRAPHY

All photomicrographs presented in this report, were prepared at a magnification of 500 diameters. The Bausch & Lomb Research Metallograph was used, with the ribbon filament light source and metallographic plates. Standard darkroom procedures were followed for development of the plates and subsequent reproduction of the contact prints.

Typical areas of Samples Nos. 1 and 2 were etched in $HF\text{-}NH_4F$ solution suggested by the Originator. This solution, at 50° C, severely attacked the aluminum while only faintly attacking the niobium. The aluminum was quickly dissolved to a level considerably lower than the niobium. As no fusion zone was in evidence, in spite of the severe attack, the Originator stated that the etch was adequate for his purposes and requested the photomicrographs presented as Figures 9 and 10 on sheet 12.

W. G. Grenier 7-29-64
(Signature) *(Date)*

NO. 1

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

DATA: Photomicrographs, Etched Condition of Samples Numbered 1 and 2

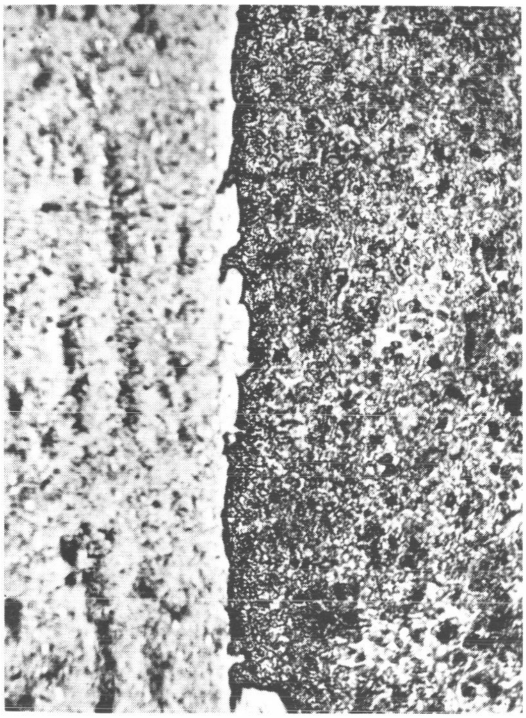


Figure 9–Sample No. 1.

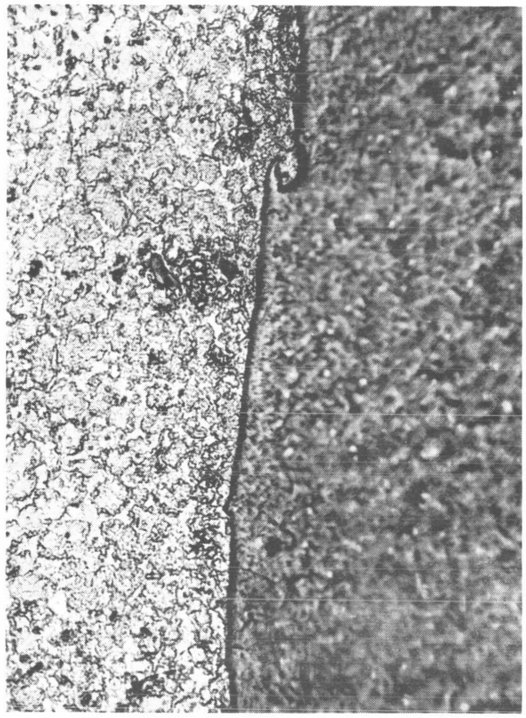


Figure 10–Sample No. 2.

Magnification: 500 diameters

$$\text{Etch:}\quad \left.\begin{array}{ll} HF & -\ 30\ \text{ml} \\ NH_4F & -\ 10\ \text{gm} \\ H_2O & -\ 60\ \text{ml} \end{array}\right\}\ \text{at } 50^\circ C$$

670-18 (2/64)

W. G. Grenier (Signature) 7-29-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S70-01	810-8

CONCLUSIONS:

The ultrasonic welding technique used in this preliminary evaluation was the "hit-or-miss" type and all welding accomplished in four hours. When a specimen was obtained that would show adherence or welding to the extent that the Nb would fail adjacent to the weld, this was called acceptable. Upon thermal cycling, this specimen proved satisfactory as a bimetallic element.

Samples of the exploratory specimens were sectioned & metallographically examined for bonding. The photomicrographs in this report show the welded samples. The two metals (Al and Nb) are in intimate contact and there appears to be a diffusion zone. But due to the time allowed, this zone could not be revealed. After pulling specimens apart and observing the interface, welding with metallurgical bonding seems to have taken place.

More welding using this technique must be performed with these two metals before definite conclusions can be drawn regarding type and extent of bonding.

C. E. Vest (Signature) 1-29-65 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
C. E. Vest	11	S-120	RAE-A	673S93-01	1700-2

DATE IN	DATE COMPLETED	PERFORMED BY
4-19-64	Interim	W. G. Grenier

NAME OF TEST

Metallurgical Examination

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Brush Be-Cu Alloy – 10, strip, 0".002 thick
Brush Be-Cu Alloy – 25, strip, 0".002 thick
Brush Be-Cu Alloy – 125, strip, 0".002 thick
Brush Be-Cu Alloy – 190, strip, 0".002 thick
Consil strip, 0".002 thick

EQUIPMENT INVOLVED:

All the equipment incorporated in a well equipped Metallographic Laboratory, including; Buehler Automet Polishing equipment, hand polishing equipment, Kentron Microhardness testor, Bausch & Lomb Research Metallograph, and associated well equipped darkroom facility.

RESULTS:

1. Grain size measurements, Table No. 3, Sheet 16.
2. Microhardness tests, Table No. 4, Sheets 18-22 inclusive.
3. Summary of Microhardness tests, Table 4, Sheet 22.
4. Alloy composition, Table No. 5, Sheet 23.
5. Photomicrographs, 500×, Sheets 24-28 inclusive.

W. G. Grenier (Signature) 6-17-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: GENERAL

Three samples of Beryllium-Copper strip and one sample of Consil strip were submitted by the Originator on 4-16-64 for Metallurgical examinations. One additional sample of Beryllium-Copper strip, silver plated on one face, was submitted on 6-4-64. Conditions for each sample, as it was received, is given in Table 1, sheet 13, of the data. All strip received was two one-thousandths of an inch in thickness.

It was desired that the microstructure be observed in each of three distinct orientations, i.e. transverse, longitudinal, and parallel to the rolled surface. For the transverse and longitudinal studies, it was necessary to clamp the thin sheet sections in sandwich form with thicker Berylco-25 strip separators. The clamping method is shown in Figure 1, sheet 14 of the data. Several different mounting mediums were evaluated for those specimens to be orientated in the plane of rolling. A glass filled epoxy mix was evolved which served this purpose. Individual specimens designations, alloys, and mounting mediums are given in Table 2, sheet 15, of the data.

It was also desired that grain size in each of the foregoing orientations be determined by some appropriate method. As the grains, in most cases, are in the strained condition and are not equiaxial, the Heyn's Intercept Method was used in every case. This method is described, in detail, on page 300 of Principles of Metallographic Laboratory Practice

W. G. Grenier (Signature) 6-16-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: GENERAL (Continued)

by G. L. Kehl, Third Edition. The results of those measurements are given in Table 3, sheet 21, of the data.

Microhardness tests were conducted using the Kentron Microhardness tester with a fifty gram load. This instrument is equipped with a Bleeker, Filar Micrometer microscope, whose hightst magnification objective is 50× with an 0.85 N.A. The hardness tests were performed on the transverse and longitudinal specimens, as these offered the greatest assurance of stability. Individual microhardness impression orientation is shown in Figures 2 and 3, sheet 17, of the data. Actual hardness test data is given in Table 4, sheets 18-22, with sheet 22 containing a summary of said data.

Mr. W. E. Tinney, Metallurgist for Brush Beryllium Corp., of Hamburg, Pa., was consulted as to the specific chemical compositional differences in the various alloys. The information so obtained is presented in Table 5, sheet 23, of the data.

Photomicrographs, showing typical structures of the submitted samples, were prepared at magnifications of 500 diameters in each of the previously detailed orientations. They are presented as Figures 4-18 inclusive, sheets 24-28, of the data. In discussions with Mr. C. Simonson, Metallographer for Brush Beryllium Corp., Hamburg, Pa., it was concluded that:

W. G. Grenier (Signature) 6-17-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: GENERAL (Continued)

1. The Gamma phase will precipitate only at the grain boundaries. At lower magnifications, (< 250×) a fine line grain boundary indicates normal gamma precipitation.

2. The Beta phase precipitates in the alpha matrix and in the grain boundaries. It will appear as a fine pepper, dispersed in the alpha. If excessive beta is precipitated in the grain boundaries, there will be less pepper effect and a reduction in strength.

3. Beryllides are a chemical combination of Cobalt and Beryllium. They appear as highly reflective irregularly shaped crystals occurring in both the alpha matrix and at the grain boundaries. They are generally more highly reflective than primary beta and must be viewed directly under the microscope to be differentiated.

4. Primary Beta, is a Beryllium rich phase precipitated during cooling of the original ingot. If the original ingot is not chilled rapidly enough, segregated primary beta may form. This would result in a weak, brittle, structure. Subsequent working and heat treatments may break-up the structure, but does not eliminate the primary beta. If excessive primary beta is present, age hardened parts may have premature failure. If primary beta stringers are present, the material should be summarily rejected.

W. G. Grenier (Signature) 6-17-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: GENERAL (Continued)

5. In alloy 10, the cobalt precipitates as true cobalt because there is insufficient Beryllium present to form beryllides.

6. Discussions on effect of grain size pointed up the desirability of maintaining grain sizes such that the average grain diameter is > 0.01 mm. That is to say, generally there should be fewer than 12,750 grains per square millimeter. With decreasing grain size it is readily apparent that there will be an increase in grain boundary area. As this area becomes excessively large there may be insufficient beryllium to form precipitates in all the grain boundaries and the alpha matrix. The effective depletion of gamma in the grain boundaries means lower ductility and strength.

7. The alpha plus beta phase is a stable condition above 1067°F, but is metastable at room temperatures. This indicates that if held at room temperature for a sufficient length of time, the beta could precipitate out of solution. If the material is heated in the range of from 600°F to 700°F this precipitation is expedited. Also, if given sufficient time at temperature, the beta phase will tend to transform to the smaller, more ordered structure of gamma. The gamma precipitates occur first at the higher energy areas of the grain boundaries. However, extended times at temperature can cause, not only the transformation of beta to gamma, but also spheroidizing and agglomeration. This will result in losses in ductility and strength.

W. G. Grenier 6-17-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: SPECIMEN PREPARATION, METALLOGRAPHIC

I. TRANSVERSE AND LONGITUDINAL SPECIMENS

1. Mounting Procedure

1.1 Those specimens numbered 1, 3, 5, and 7, were clamped in mild steel clamps and mounted as shown in Figure 1 of the data. Specimen number 9 was clamped in a Berylco-25 alloy clamp and mounted in green bakelite. In the case of specimen number 9 a piece of material was mounted with the specimen, on the transverse side, to act as a gauge mark.

1.2 To assure tightness of clamps; it is recommended that, with the specimen material between them, they be compressed in a machinists vise while tightening the screws. This will minimize possibilities of voids between layers, which could trap foreign matter.

2. Polishing Procedures

2.1 Specimens were rough ground on the Buehler wet belt surfacer; using an 80 grit silicon carbide belt, copious quantities of water, and a very light hand pressure. Rough grinding was continued until a smooth surface was obtained over the entire specimen.

W. G. Grenier (Signature) 6-17-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: SPECIMEN PREPARATION (Continued)

2.2 Specimens were rough polished through the 600 grit silicon carbide papers using the Buehler three wheel slow speed polishing table. This table is equipped with an Automet attachment for each wheel. For each grit, the load setting was 20 pounds and the time of polishing was 3 minutes. Copious quantities of water were used in every case. Subsequent to the 600 grit paper, the specimens were polished using 15 micron sized diamond compound followed by 6 micron sized·diamond. Diamond polishing was accomplished with Texmet cloth, 20 pound load setting and the 3 minute polishing time.

2.3 Stress cracks, similar to those apparent in Figure 1, were observed in the lucite mounts upon removal from the 320 grit paper. At this time they were not considered severe enough to cause difficulties. However subsequent to the 6 micron diamond polishing they appeared quite severe. Therefore each specimen was replaced in the Powermet mounting press. A small quantity of Transoptic powder was added to the mount. Each specimen was held at temperature and slightly reduced pressure for 1/2 hour. Upon removal from the mold, all specimens appeared to be in excellent condition. Stress cracking of this type is rarely encountered when bakelite is used as the mounting medium.

2.4 It was felt that pressure exerted by the Automet clamping ring might be a contributing factor to the above mentioned stress cracks. Therefore each specimen was

W. G. Grenier (Signature) 6-17-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: SPECIMEN PREPARATION (Continued)

finish polished by hand. The two wheel hand polishing table was used for this operation. A slurry of 600 grit aluminum oxide and tap water was used with billiard cloth on the first, fast wheel. A slurry of Fisher's Gamal alumina and deionized water with a well dressed microcloth was used for the final polish. A very light hand pressure was used throughout.

3. Etching Procedures

3.1 For etching the Beryllium Copper alloys, to point up precipitates and general structure, an ammonium presulfate etchant was used, as follows:

$(NH_4)_2 S_2 O_8$ – 20 gms

$NH_4 (OH)$ – 30 ml

H_2O_2 (30%) – 10 ml

H_2O (D.I.) – 140 ml

This etchant should be mixed fresh each day, and discarded when finished with a specific series.

3.2 The ammonium presulfate etch, while delineating constituents tends to stain the specimen. Therefore a second etchant of dilute acetic acid solution was used to enhance contrast, for grain measuring purposes.

W. G. Grenier (Signature) 6-17-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: SPECIMEN PREPARATION (Continued)

3.3 It had been recommended that the Consil alloy be treated as pure silver, since it was 98% silver. Therefore a potassium cyanide and ammonium persulfate etch was used. This resulted in severe pitting and staining. The specimen was repolished and etched using a silver alloy etchant, as follows:

NH_4 (OH) – 100 ml

H_2O_2 (30%) – 40 ml

This etchant produced the results given in the data.

3.4 Repolishing and etching follows no set rule and each specimen is worked on individually. This procedure, of necessity, is dependent on that individual who is preparing the photomicrograph and may vary until he is certain that the true structure is clearly revealed.

II. PLANE, PARALLEL TO ROLLING PLANE, SPECIMENS

1. Mounting Procedure

1.1 Specimens orientated in this manner were numbered with the even numbers 2-10 inclusive. A small section was cut from the original sample and two edges bent upward to act as anchors in the mounting medium. It was known that this type of specimen, 0.002 in thickness, would not adhere to bakelite, but it was felt that it might adhere to lucite.

W. G. Grenier (Signature) 6-17-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: SPECIMEN PREPARATION (Continued)

Therefore attempts were first made to mount specimens in lucite. It was learned that there was no effective adhesion between the lucite and the thin metal strip. Buehler cold mounting plastic was then tried without success.

1.2 Due to lack of adherence in the foregoing it was decided to use a glass filled epoxy mix. This procedure while frequently producing excellent mounts, has given difficulty in the past. The problem is in the hardening or curing process. It seems that regardless of the care taken in preparation of the mix, it frequently fails to harden properly. Failure to harden occurred again in this instance so new specimens were prepared using different mixes until the following was arrived at:

Hysol 2038 epoxy	– 60 gms
325 mesh, ground glass	– 20 gms
Hysol 3404 hardener	– 8.2 gms

Blend the glass and epoxy thoroughly and allow to set for a minimum of 24 hours. This permits trapped air to escape. Blend the hardener, thoroughly with the glass and epoxy, stirring for at least three minutes. With specimens located in bakelite ring forms, pour blended mix over and around each specimen until the ring form is filled. Permit mount to remain stationary for a minimum of 24 hours to harden thoroughly.

W. G. Grenier 6-17-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: SPECIMEN PREPARATION (Continued)

2. Polishing Procedures

2.1 Each specimen was rough ground on the wet belt surfacer, using a 120 grit silicon carbide belt and moderate flow of water. This operation was continued only long enough to remove any excess epoxy which was lodged on the face of the specimen. Frequent checks were required and a very gentle pressure as the specimens were very thin and could easily be ground completely away. With the first indication of contact between a specimen and the abrasive belt, rough grinding was discontinued.

2.2 Rough polishing, through the 600 grit paper, was accomplished on the Buehler Handimet hand polisher. Each specimen was rotated 90° with each decreasing grit size. A very light hand pressure was maintained throughout and polishing continued just long enough to remove most of the scratches from the preceding polishing operation.

2.3 Final polishing was identical to that described under Article I, subparagraph 2.4, sheet 7 of this report.

W. G. Grenier 6-17-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

PROCEDURE: SPECIMEN PREPARATION (Continued)

3. Etching Procedures

3.1 Etching procedures, with etchants used were identical to those described in Article I, paragraph 3, sheets 8 and 9 of this report.

III. PREPARATION OF PHOTOMICROGRAPHS

1. Photomicrographs were prepared of all specimens at magnifications of 500 diameters. The Bausch & Lomb Research metallograph, with bright field illumination was used with the 41X-0.65 N.A. objective lens, in conjunction with the 10X negative amplifier, and bellows draw of 25.8 centimeters. Kodak metallographic plates were used to record the image. Standard darkroom procedures were followed for reproduction of the finished photomicrographs presented herein.

W. G. Grenier (Signature) 4-23-64 & 5-22-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA:

Table 1
INFORMATION RECEIVED WITH MATERIAL

SAMPLE NO.	MATERIAL	HEAT	TEMPER	NOMINAL T.S. 1000 P.S.I.	CONDITION RECEIVED
1	Brush Alloy-125	1308-03-13	XHMS	175	Flat Sheet, 2" × 3'
2	Brush Alloy-190	76621	XHMS	175	Flat Sheet, 2" × 3'
3	Brush Alloy-10	76622	HT	112	Flat Sheet, 2" × 3'
4	CONSIL	As Received From DeHavilland – Tubular			
5	Brush Alloy-25	As Received From DeHavilland – Tubular			

670-19 (2/64)

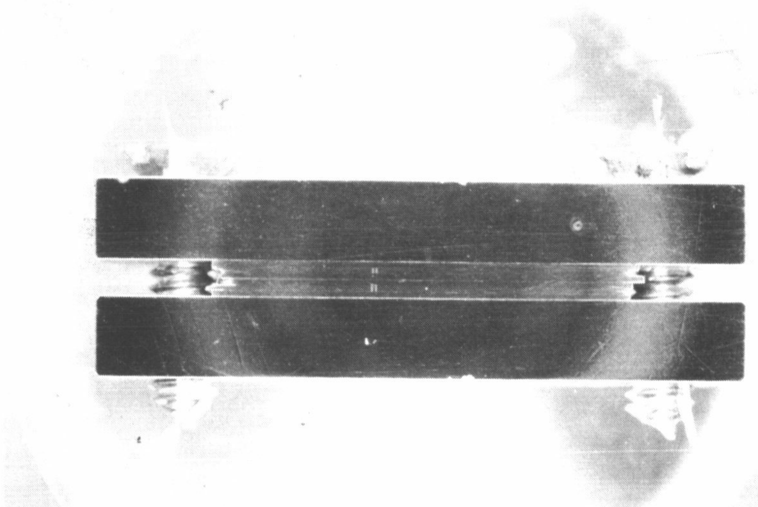
W. G. Grenier (Signature)

4-23-64 & 5-22-64 (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA: Photomacrograph of Typical Specimen Mount



3½X

Figure 1–Specimen Sandwich, Mounted in Lucite

Shows special clamps, developed by personnel of the Structural and Mechanical Applications Section for holding thin metallurgical specimens.

In the typical sandwich shown, specimens of transverse and longitudinal sections 0".002 thick, are separated by annealed Berylco-25 filler strips 0".010 thick. The specimen sandwich can be mounted in any appropriate medium.

670-18 (2/64)

W. G. Grenier (Signature) 4-16-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA:

Table 2
METALLOGRAPHIC SPECIMEN DESIGNATIONS

SPECIMEN NO.	SAMPLE NO. FROM TABLE 1	ALLOY	SPECIMEN DESCRIPTION NOTE	SPECIMEN DESCRIPTION MOUNTING MEDIUM
1	1	125	*	Lucite
2	1	125	**	Glass Filled Epoxy
3	2	190	*	Lucite
4	2	190	**	Glass Filled Epoxy
5	3	10	*	Lucite
6	3	10	**	Glass Filled Epoxy
7	4	Consil	*	Lucite
8	4	Consil	**	Glass Filled Epoxy
9	5	25	*	Green Bakelite
10	5	25	**	Glass Filled Epoxy

Specimen Description Notes:

*0".002 thick, transverse and longitudinal cross sections, sandwiched with annealed Berylco-25, 0".010 thick sheet as separators. The sandwich was clamped in special, "Thin Metal Clamps," and mounted in the designated medium.

**0".002 thick strip; orientated, as nearly as practical, such that the observed surface will be a plane parallel to the rolled surface.

670-19 (2/64)

W. G. Grenier (Signature) ~~4-23-64~~ & 5-22-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA:

Table 3
AVERAGE GRAIN SIZES—AS DETERMINED BY HEYN'S METHOD

SPECIMEN NO.	ALLOY	SPECIMEN ORIENTATION	AVERAGE GRAIN SIZE NO. OF GRAINS PER SQ. MM	LENGTH X 10^{-3}	WIDTH X 10^{-3}
1	125	Transverse	4,100	18	13
1	125	Longitudinal	3,300	25	12
2	125	Parallel Rolled Face	2,600	22	17
3	190	Transverse	8,900	13.6	8.3
3	190	Longitudinal	6,600	16	9.5
4	190	Parallel Rolled Face	3,400	19.5	15
5	10	Transverse	14,500	10.6	6.7
5	10	Longitudinal	14,000	13	5.7
6	10	Rolled Face	15,000	8	8.3
7	Consil	Transverse	1,100	45	20
7	Consil	Longitudinal	2,700	23	16.3
8	Consil	Rolled Face	900	35	32
9	25	Transverse	2,100	25	19
		Longitudinal	2,030	31	16
		Rolled Face	1,600	28	22

Grain size based on average of 6 measurements per section

670-19 (2/64)

W. G. Grenier (Signature) 6-8-64 (Date)

NO. I

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA: Microhardness Impression Orientation.

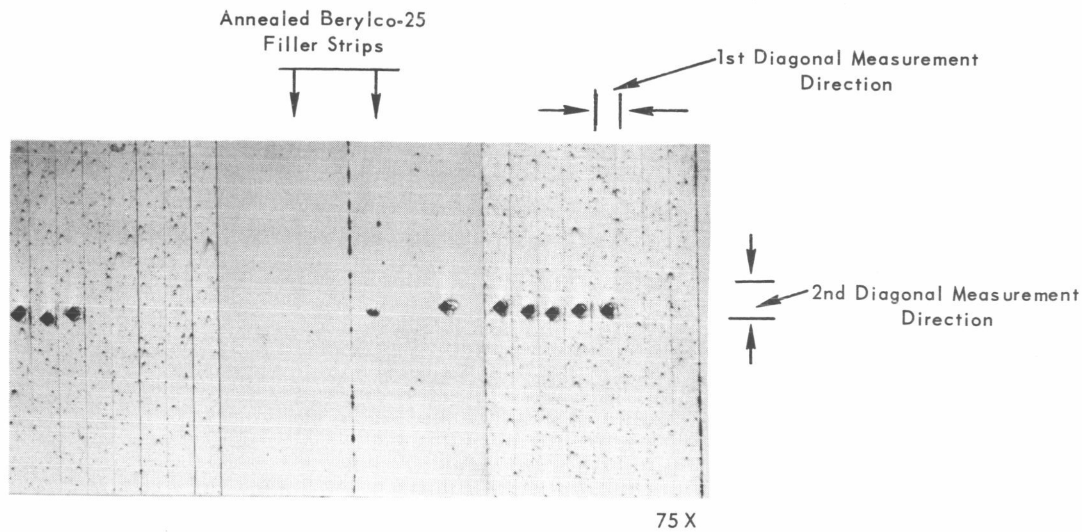


Figure 2

Typical of microhardness impressions on polished Consil alloy

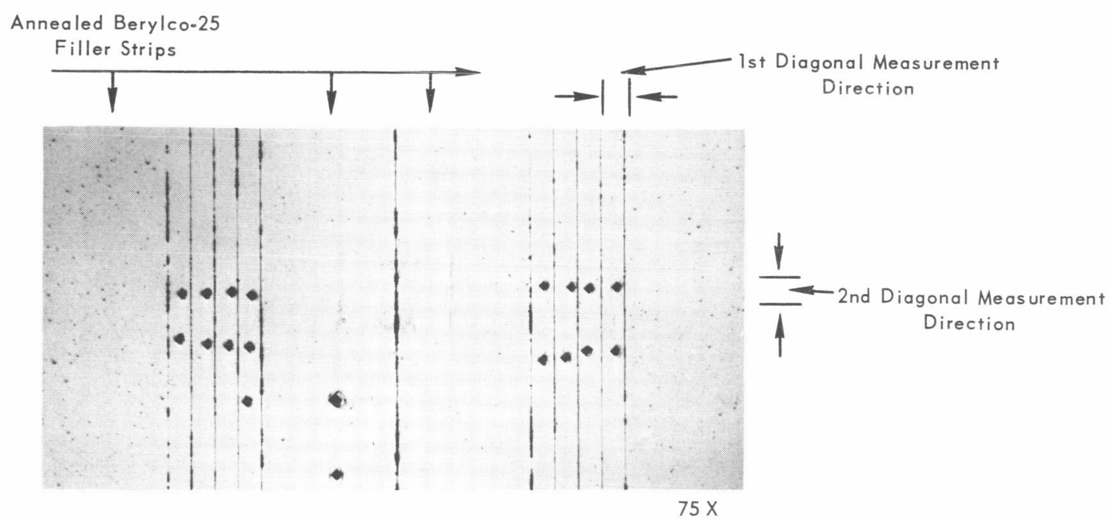


Figure 3

Typical of microhardness impressions on polished Be-Cu alloys.

670-18 (2/64)

W. G. Grenier (Signature) 6-8-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA:

Table 4
MICROHARDNESS TEST DATA

LOAD = 50 gms.
OBJ. = 50X, 0.85 N.A.

SPECIMEN NO.	ALLOY	ORIENTATION	DIAGONAL LENGTHS FILAR UNITS 1ST	2ND	AVG.	MICRONS AVG.	TABLE VALUE	D.P.H.N.	CONVERT TO ROCKWELL
1	125	Transverse	85	91	88	17.7	5.92	296	C-29
			86	94	90	18.06	5.66	283	28
			89	92	90	18.07	5.66	283	28
			89	91	90	18.07	5.66	283	28
			89	90	90	18.07	5.66	283	28
			83	90	87	17.46	6.06	303	30
			84	86	85	17.07	6.34	317	32
			88	90	89	17.87	5.79	289	28
			88	92	90	18.07	5.66	283	28
			88	95	91	18.26	5.54	277	27
						AVERAGE		289.7	C-28.6
		Longitudinal	86	90	88	17.65	5.92	296	C-29
			84	91	88	17.65	5.92	296	29
			86	89	87	17.46	6.06	303	30
			86	88	87	17.46	6.06	303	30
			87	84	86	17.26	6.20	310	31
			89	86	88	17.65	5.92	296	29
			88	94	91	18.26	5.54	277	27
			87	92	89	17.87	5.79	289	C-28
1	125					AVERAGE		296	C-29

670-19 (2/64)

W. G. Grenier (Signature) 5-25-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA:

Table 4
MICROHARDNESS TEST DATA (Continued)

SPECIMEN NO.	ALLOY	ORIENTATION	DIAGONAL LENGTHS FILAR UNITS 1ST	2ND	AVG.	MICRONS AVG.	TABLE VALUE	D.P.H.N.	CONVERT TO ROCKWELL
3	190	Transverse	93	95	94	18.86	5.19	259	C-24
			89	94	92	18.48	5.42	271	26
			95	94	94	18.86	5.19	259	24
			91	98	94	18.86	5.19	259	24
			95	94	94	18.86	5.19	259	24
			94	95	94	18.86	5.19	259	24
			88	92	90	18.07	5.66	283	C-28
						AVERAGE		267	C-25
		Longitudinal	87	88	88	17.65	5.92	296	C-29
			95	93	94	18.86	5.19	259	24
			88	95	92	18.48	5.42	271	26
			88	90	89	17.87	5.79	289	28
			92	88	90	18.07	5.66	283	28
			89	93	91	18.26	5.54	277	27
			89	93	91	18.26	5.54	277	27
			91	94	92	18.48	5.42	271	C-26
3	190					AVERAGE		279	C-27

670-19 (2/64)

W. G. Grenier (Signature) 5-25-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	OGO	673S93-01	1700-2

DATA:

Table 4
MICROHARDNESS TEST DATA (Continued)

SPECIMEN NO.	ALLOY	ORIENTATION	DIAGONAL LENGTHS FILAR UNITS 1ST	2ND	AVG.	MICRONS AVG.	TABLE VALUE	D.P.H.N.	CONVERT TO ROCKWELL
5	10	Transverse	103	109	106	21.3	4.09	205	B-93
			101	110	104	20.9	4.25	212	94
			99	104	102	20.5	4.42	221	96
			100	103	101	20.3	4.50	225	97
			97	100	99	19.87	4.68	234	99
			100	101	100	20.08	4.59	230	98
			100	104	102	20.50	4.42	221	96
			99	103	101	20.3	4.50	225	B-97
						AVERAGE		222	B 96
		Longitudinal	97	104	101	20.3	4.50	225	B-97
			98	101	100	20.1	4.59	230	98
			100	101	100	20.1	4.59	230	98
			97	101	99	19.87	4.68	234	98
			98	104	101	20.3	4.50	225	97
			97	101	99	19.87	4.68	234	98
			96	103	100	20.1	4.59	230	98
			99	103	101	20.3	4.50	225	B-97
5	10					AVERAGE		229	B-97.5

670-19 (2/64)

W. G. Grenier (Signature) 5-25-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA:

Table 4
MICROHARDNESS TEST DATA (Continued)

SPECIMEN NO.	ALLOY	ORIENTATION	DIAGONAL LENGTHS FILAR UNITS 1ST	2ND	AVG.	MICRONS AVG.	TABLE VALUE	D.P.H.N.	CONVERT TO ROCKWELL
7	Consil	Transverse	132	128	130	26.1	2.72	136	B-74
			137	140	138	27.7	2.42	121	68
			135	133	134	26.9	2.56	128	71
			135	131	133	26.7	2.60	130	72
			138	135	136	27.3	2.49	125	B-70
						AVERAGE		128	B-71
		Longitudinal	133	138	135	27.1	2.53	126	B-70
			141	141	141	28.3	2.32	116	65
			140	140	140	28.1	2.35	118	67
			143	145	144	28.9	2.22	111	62
			130	138	134	26.9	2.56	128	B-71
7	Consil					AVERAGE		118	B-67
Filler Strips	BERYLCO-25, annealed		–	–	122	24.48	3.09	160	B-83
			–	–	117	23.48	3.36	168	85
			–	–	116	23.28	3.42	171	85
			–	–	119	23.9	3.25	163	83
			–	–	120	24.1	3.19	160	B-83

670-19 (2/64)

W. G. Grenier (Signature) 6-8-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA:

Table 4
SUMMARY

SPECIMEN NO.	ALLOY	ORIENTATION	HARDNESS 50 GRAM D.P.H.N.	D.P.H.N. CONVERT TO ROCKWELL
1	125	Transverse	290	C-29
1	125	Longitudinal	296	C-29
3	190	Transverse	267	C-25
3	190	Longitudinal	279	C-27
5	10	Transverse	222	B-96
5	10	Longitudinal	229	B-98
7	Consil	Transverse	128	B-71
		Longitudinal	118	B-67

In accordance with Brush Beryllium Co. Data Sheets, the hardness for specific alloys and tempers should be as follows:

Alloy	Temper	Rockwell Hardness
190	XHMS	30N of 56-61, converts to C of 36-42
10	HT	30T of 79-82, converts to B of 95-100

670-19 (2/64)

W. G. Grenier (Signature) 6-4-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA:

Table 5
CHEMICAL COMPOSITION OF ALLOYS IN PERCENT

ALLOY	% Be	% Co	% Cu	% Ni
10	0.40-0.70	2.35-2.70	Balance	–
25	1.80-2.05	0.20-0.30	Balance	–
125	1.80-2.05	< 0.19	Balance	0.05 max
190	1.87 min-2.05 max, Co and/or Ni 0.20 min, Cu + Be + additives = 99.5 min.			

Notes: Alloy 25 is common alloy, not as closely controlled as others.

Alloy 125 = very closely controlled alloy-25, with residuals purportedly, virtually eliminated.

Alloy-190 = modification of alloy-25

Consil → 98% Ag, 2% Mg + Ni + Residuals

670-19 (2/64)

W. G. Grenier (Signature) 5-26-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA: Alloy-10, Photomicrographs, 500 Magnifications

Typical Structures

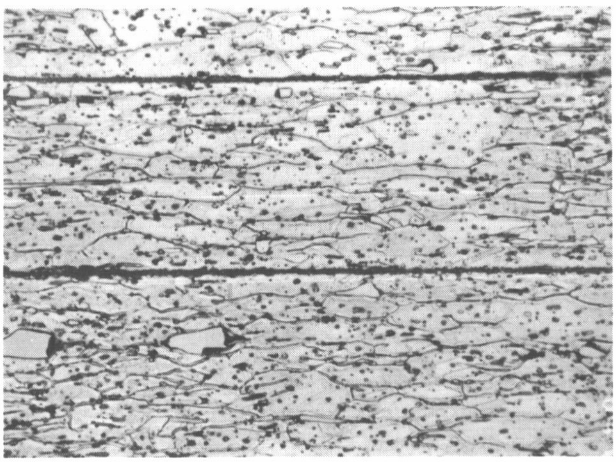


Figure 4–Transverse

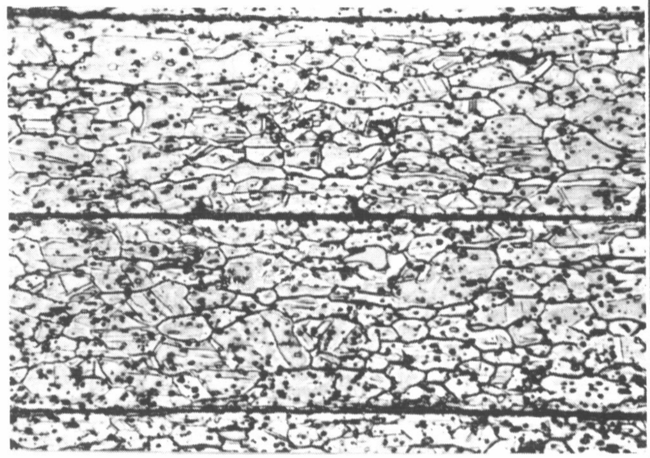


Figure 5–Longitudinal

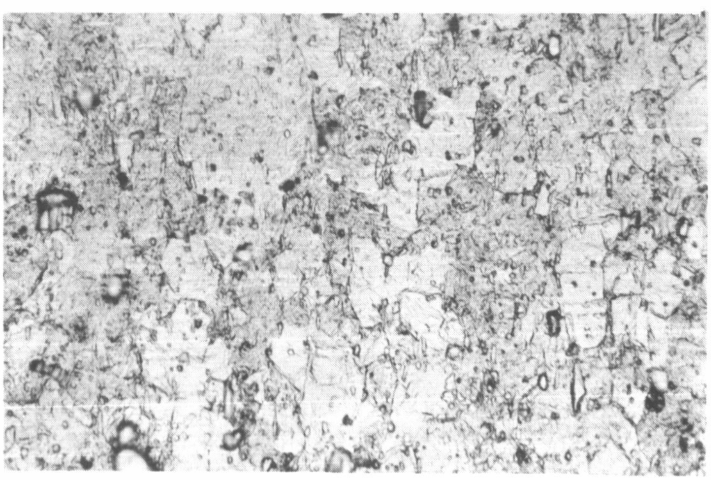


Figure 6–Plane Parallel to Rolling Plane

Figures 4 and 6 show Free Cobalt precipitate, in predominantly stringer form. Be is in solid solution with Cu.

Etchant:

$(NH_4)\ S_2O_8$	20 gms
$NH_4\ OH$	30 ml
H_2O_2 (30%)	10 ml
H_2O	140 ml

670-18 (2/64)

W. G. Grenier (Signature) 5-17-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA: Typical Structures, Alloy 125, Photomicrographs, 500 Magnifications

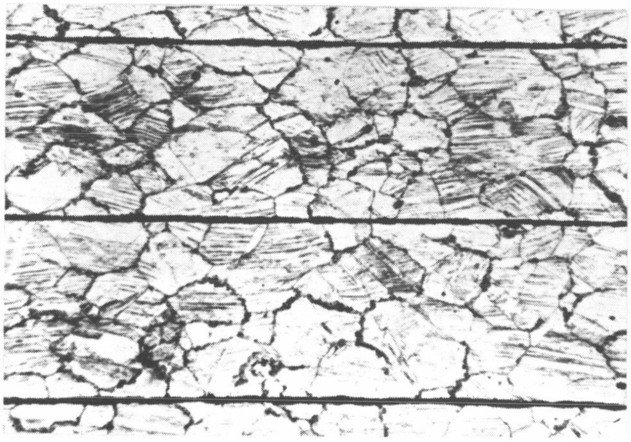


Figure 7–Transverse

Figure 8–Longitudinal

Shows mechanical twinning, strain hardening, gamma precipitate in grain boundaries.

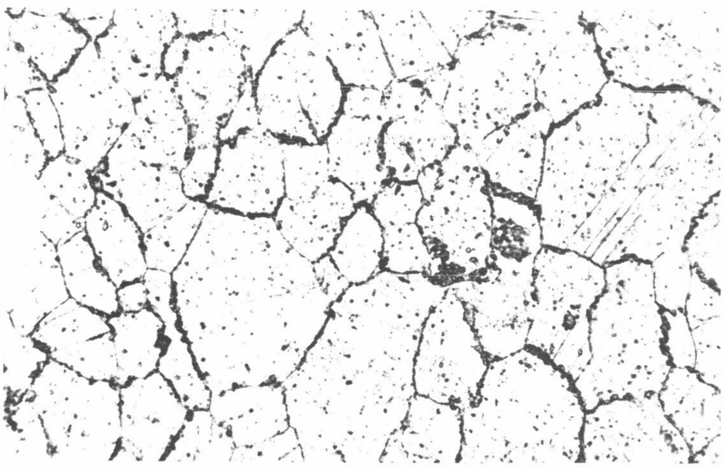


Figure 9–Plane Parallel to Rolling Plane.

Etchant:

Ammonium Persulfate etch outlined on sheet 24 of the data, followed by dilute acetic acid to enhance contrast for grain measurement.

Pepper spots are Beta precipitate in alpha matrix.

670-18 (2/64)

W. G. Grenier (Signature) 5-18-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA: Typical Structures, Alloy 190, Photomicrographs, 500 Diameters

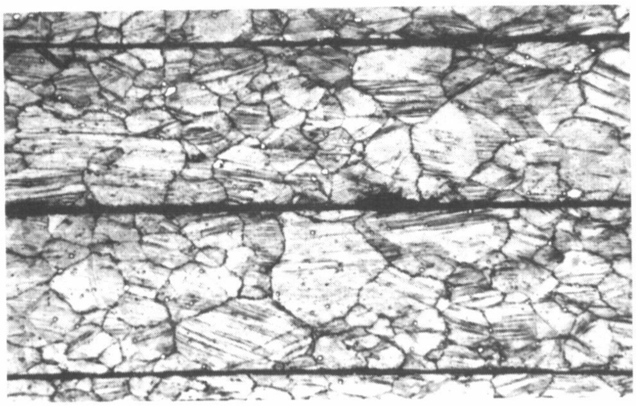


Figure 10–Transverse

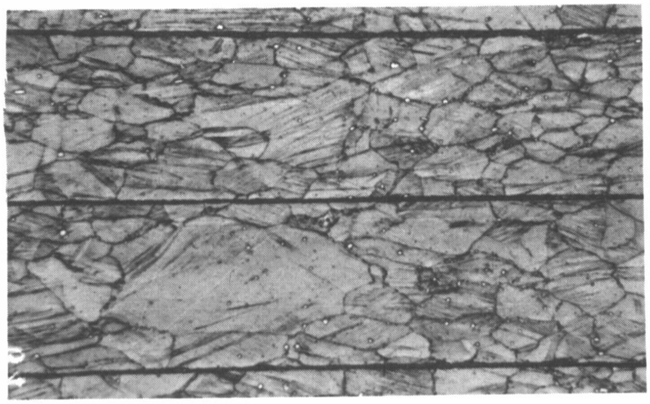


Figure 11–Longitudinal

White irregularly shaped spots are Cobalt Beryllides with some primary Beta in the form of spheroids. Also shows cold-work, and some Beta precipitate in the alpha matrix.

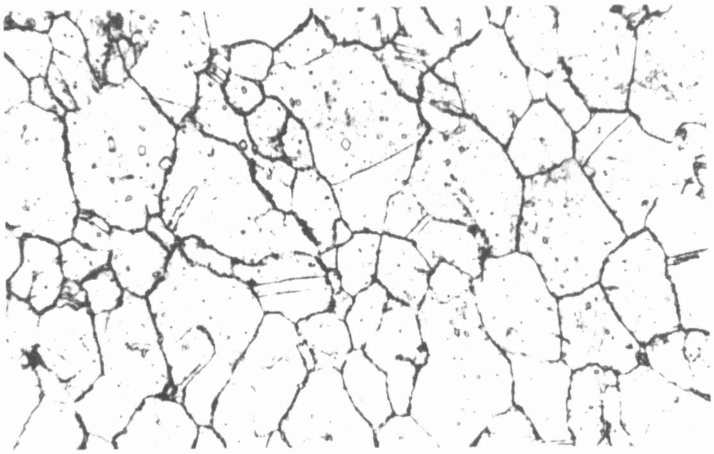


Figure 12–Plane Parallel to Rolling Plane.

Etchant:

Ammonium Persulfate etch, outlined on sheet 24 of the data. Figure 12 was subsequently etched in dilute acetic acid to enhance contrast for grain size determination.

Persulfate etch followed by dilute acetic points up Beryllides, some Beta precipitate, and mechanical twinning.

670-18 (2/64)

W. G. Grenier (Signature) 5-8-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA: Typical Structures, Alloy-Consil; Photomicrographs, 500 Magnifications

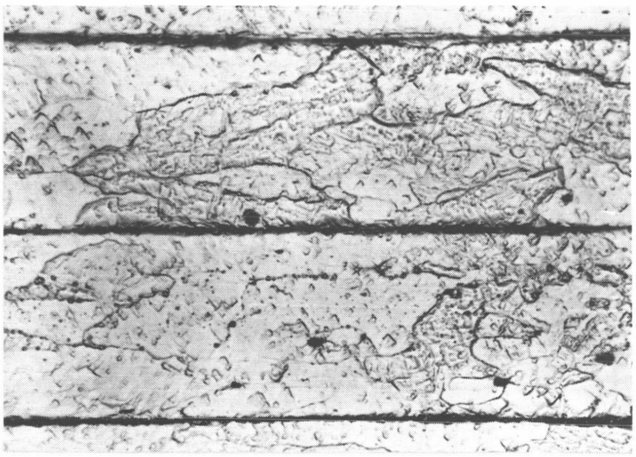


Figure 13–Transverse

Figure 14–Longitudinal

Etchant:

NH_4OH . . . . 100 ml

H_2O_2 (30%) . . . . 40 ml

Figure 15–Plane Parallel to Rolling Plane.

670-18 (2/64)

W. G. Grenier (Signature) 5-18-64 (Date)

NO. I

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

DATA: Typical Structures, Alloy-25, Photomicrographs, 500 Magnifications

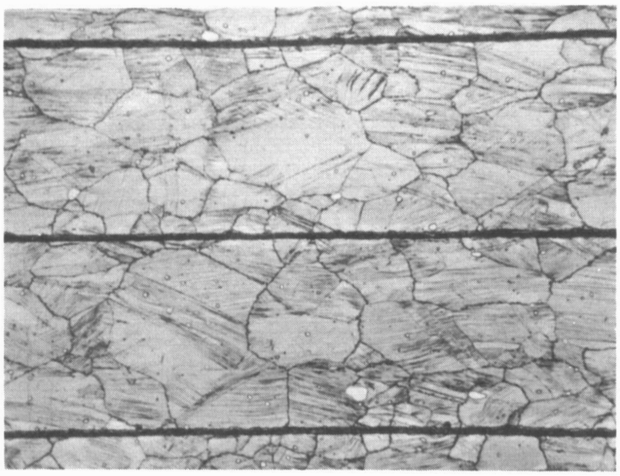


Figure 16–Transverse

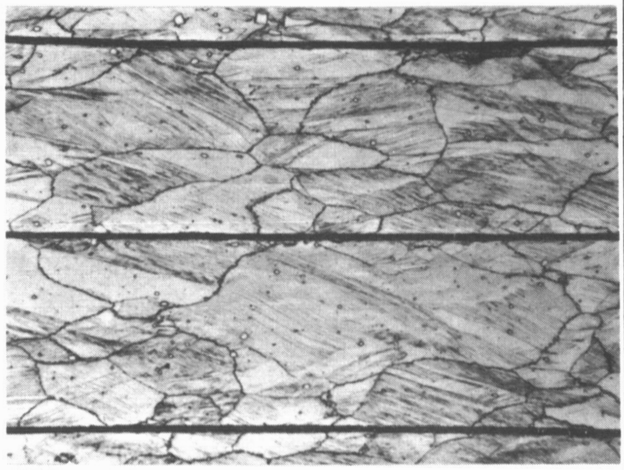


Figure 17–Longitudinal

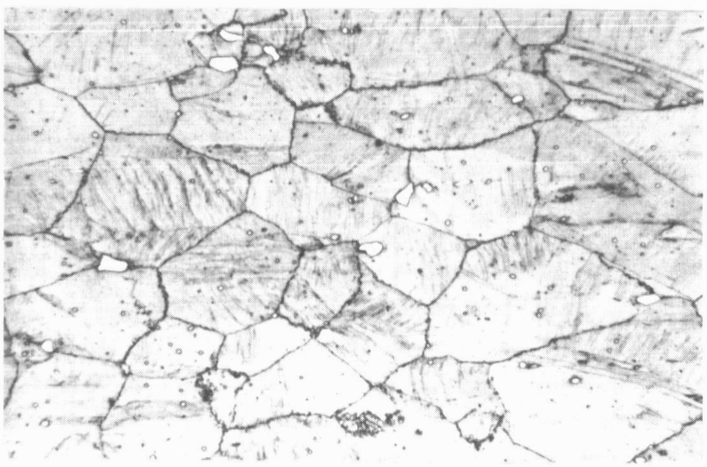


Figure 18–Plane Parallel to Rolling Plane.

Etchant:

Ammonium Persulfate etch as outlined on sheet 24 of the data

Figures show Beta precipitate in alpha matrix in form of pepper like particles, plus irregularly shaped Beryllides. Gamma is precipitated at grain boundaries and some twinning and work hardening is in evidence.

670-18 (2/64)

W. G. Grenier (Signature) 6-8-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE-A	673S93-01	1700-2

CONCLUSIONS:

The purpose of this evaluation was to determine: (1) grain size, (2) micro hardness, and (3) to show typical microstructure which would point up any abnormalities.

The grain size, hardness, and microstructures are typical (according to published literature) for the metallurgical condition of the alloys. The microstructures show no abnormalities. The photomicrographs and specimen preparation techniques will be useful for additional work on these alloys.

C. E. Vest (Signature) 6-18-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
C. E. Vest	11	S-120	RAE	673S93-03	1700-4

DATE IN	DATE COMPLETED	PERFORMED BY
9-17-64	9-30-64	W. G. Grenier

NAME OF TEST

Metallographic Examination

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Four samples of Cadmium plated Be Cu sheet were examined metallographically to determine Cd diffusion into the Be Cu substrate.

EQUIPMENT INVOLVED:

Metallurgical thin specimen clamps of Be Cu-25 alloy, Powermet mounting press, Green Bakelite, Rough belt Surfacer, Handimet hand polisher, Buehler slow speed polishers with Automet attachments, Buehler fast and slow speed hand polishing table, ultrasonic cleaning apparatus, Metallurigical bench microscopes, Bausch & Lomb Research Metallograph, various chemical reagents and hand tools, and a well equipped photographic darkroom.

RESULTS:

No clear evidence of cadmium diffusion into the Be Cu-190 alloy was observed.

W. G. Grenier (Signature) 9-30-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE	673S93-03	1700-4

PROCEDURE: SPECIMEN SELECTION AND MOUNTING

Four (4) samples of Be Cu alloy 190, as described on sheet 1 of Originator's request, were submitted for Metallographic examination. Each sample was sectioned such that both transverse and longitudinal cross sections could be examined. As the strips were very thin, they were mounted, first in metallurgical thin metal clamps and this assembly was then mounted in Green Bakelite, using the Powermet mounting press.

Specimen designation is as follows:

Specimen No. 1 – Sample No. 1, longitudinal section

Specimen No. 2 – Sample No. 1, transverse section

Specimen No. 3 – Sample No. 2, longitudinal section

Specimen No. 4 – Sample No. 2, transverse section

Specimen No. 5 – Sample No. 3, longitudinal section

Specimen No. 6 – Sample No. 3, transverse section

Specimen No. 7 – Sample No. 4, longitudinal section

Specimen No. 8 – Sample No. 4, transverse section

W. G. Grenier 9-25-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE	673S93-01	1700-4

PROCEDURE: GRINDING AND POLISHING

There are four (4) grinding and polishing stages; Rough grinding, fine grinding, rough polishing, and final polishing.

Rough grinding is accomplished on the wet belt surfacer with an eighty grit silicon carbide belt. For this operation, each specimen is ground separately and held by hand. When a uniformly smooth, flat, surface is attained, the specimen is ready for fine grinding. All subsequent grinding and polishing operations, except polishing between etches, is accomplished on the three (3) wheel polishing table. Each wheel on this table is equipped with an Automet attachment and is operated at the low speed setting.

Fine grinding and the first phase of rough polishing is done using adhesive backed Silicon Carbide abrasive papers. Free flowing tap water is used as the coolant and lubricant. Successively finer grit papers are used, starting with 180 grit, followed by; 240 grit, 320 grit, 400 grit, and 600 grit. Four hundred grit, is frequently considered to be the start of the rough polishing stage. The Automet load setting is held constant, at forty (40) pounds, through the 320 grit and dropped to thirty (30) pounds for the 400 grit and 600 grit papers. The grinding and polishing times decrease with the increasing fineness of the grit. The specimens are ground for; five (5) minutes on the 180 grit paper, four (4) minutes on the 240 grit paper, three (3) minutes on the 320 grit paper, two and one-half (2-1/2) minutes each on the 400 and 600 grit papers. Between each of the above,

W. G. Grenier (Signature) 9-28-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE	673S93-01	1700-4

PROCEDURE: GRINDING AND POLISHING (Continued)

grinding and polishing operations, the specimens are cleaned ultrasonically, in a Triton X-100 and tap water solution, for a period of two (2) minutes. This is followed by a thorough rinse in warm, free flowing, tap water. Subsequent to this rinse, following the 600 grit rough polish, the specimens are thoroughly dried using Propanol and a warm air blast.

Rough polishing is completed on a texmet cloth, charged with a six micron particle size diamond paste. A. B. Buehler lapping oil is used as the lubricant and extender. The Automet pressure setting is thirty (30) pounds, for a polishing time of two and one-half (2-1/2) minutes. The specimens are cleaned ultrasonically, for two (2) minutes in a trichloroethylene bath, followed immediately by a thorough rinse in Propanol. The specimens are then dried in a dry, warm air blast.

Final polish is accomplished, using Fisher's Gamal, suspended in de-mineralized water, on a well dressed Microcloth covered wheel. The Automet pressure is set at thirty (30) pounds and plishing time is three (3) minutes. Final cleaning is the same as that following the 600 grit rough polishing step.

At this point, the specimens are removed from their respective Automet holders, rinsed again in Propanol and dried in a dry, warm, air blast.

W. G. Grenier 9-28-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE	673S93-01	1700-4

PROCEDURE: POLISHING (Concluded)

Polishing between etchings is done by hand, on the individual specimens as required. Hand polishing is accomplished on the two wheel hand polishing table. The slow speed wheel is equipped with a well dressed Microcloth and charged with A. B. Buehler, Gamma Polishing Alumina No. 3. Demineralized water is used as the extender and lubricant. The wheel is operated at slow speed, for that length of time deemed necessary by the technician. Generally each successive polishing step will be shorter than the preceding one, while each successive etching stage will be longer.

Following final polish the Originator was notified. He scrutinized the several specimens at magnification of from 1000 diameters, to in excess of 2000 diameters. The suggested etchants were each tried with best results being obtained from a solution of Ammonium Persulfate, Ammonium Hydroxide, Hydrogen Peroxide, and distilled water. Best polishing results were obtained when a small quantity of this etchant was added to the abrasive on the final wheel.

At the Originators request, no photomicrographs were prepared.

W. G. Grenier 9-30-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	RAE	673S93-03	1700-4

CONCLUSIONS:

This metallographic examination was performed to discern if the cadmium plate on a Be Cu alloy would diffuse into the grain boundaries of the alloy thereby causing embrittlement and deterioration of the mechanical properties of the material. This evaluation is related to a project on RAE boom material as tests are underway to evaluate the use of Cd as a possible damping material.

The metallographic examination showed no discernable diffusion of the Cd into the Be Cu alloy on any of the samples. All samples had been stored for varied times at room temperature.

C. E. Vest (Signature) 10-1-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
F. Federline	11	S-126	Spin Control	673Y19-03	1200-62

DATE IN	DATE COMPLETED	PERFORMED BY
3-12-64	4-27-64	W. G. Grenier and D. F. Zbrosky

NAME OF TEST

Cone Angle Measurement System

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Method demonstrated for cone angle measurement with permanent record.

EQUIPMENT INVOLVED:

Light projectors, mirrors, reticles, tripod, camera, and miscellaneous hand tools and lenses.

RESULTS:

The system was developed to a point satisfactory to the Originator.

W. G. Grenier — 6-19-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

PROCEDURE: GENERAL

It was desired to establish and demonstrate a system for obtaining a permanent record of cone angle versus time, accurate to within 0.10 degrees of arc. The cone angle was to be recorded for angles up to, and including, 12° of arc in any direction. It was suggested that the recording device be a time synchronized movie camera. The Spin Table, will be mounted on an air bearing so the angle to be determined is from the horizontal.

APPROACH NO. 1

It was suggested that a bulls-eye be inscribed on a ground plexiglass screen and located above the air bearing table. A front surface mirror was to be mounted in the geometrical center of the table and a prism alligned with the mirror but situated above the plexiglass screen. A beam of light was to be projected through the prism onto the front surface mirror. The light from the front surface mirror would form a trace of light on the plexiglass screen and this trace was to be recorded by a time synchronized movie camera.

This approach introduced errors in front to back motion of ball measurements, as well as extensive mounting and alignment difficulties. Therefore other approaches will be tried.

W. G. Grenier 3-12-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

PROCEDURE: GENERAL (Continued)

APPROACH NO. 2

It was felt that there was a possibility that a phosphorescent tipped rod, mounted in the geometric center of the table and extending nearly to the plexiglass reticle could be employed. Preliminary calculations were based on a minimum reticle circle of 0".125 radius, for readability, to be equal to 0.2° of tilt from the horizontal. These calculations indicate that the shortest usable rod would be 35.75" in length. It was determined that this length of rod would permit excessive whip effect at 20 rpm. Therefore the required accuracy could not be maintained.

APPROACH NO. 3

This consisted of focusing a point of light, from somewhere in space, directly on a front surface mirror mounted in the table center. Reflections showed that it would be good only for one family of planes rotating about a single axis, or would require a great number of light sources.

APPROACH NO. 4

4.1 The schematic for this approach is detailed in Figure 1, sheet 5. From trigonometry it can be shown that the angle $\theta = \alpha/2$, therefore $r = d \tan 2\theta$. It was

W. G. Grenier (Signature) 3-13-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

PROCEDURE: GENERAL (Continued)

determined that the minimum readable radius would be, $r = 0.''063$. Discussions with the Originator, indicated that it would not be required to inscribe a grid with the 0.1° indicating lines. He indicated that lines scribed at 0.2° indications would be adequate with estimations of the readings to be taken between them.

4.2 Lengthy discussions and some experimentation, with this approach, was held with personnel of the Fabrication Division–Optical Shop. The conclusion was that:

4.2.1 While this approach is feasible there are several problems to be solved.

4.2.2 The approach requires a columnator for adequate focusing of the light source.

4.2.3 A small but high intensity light source is mandatory.

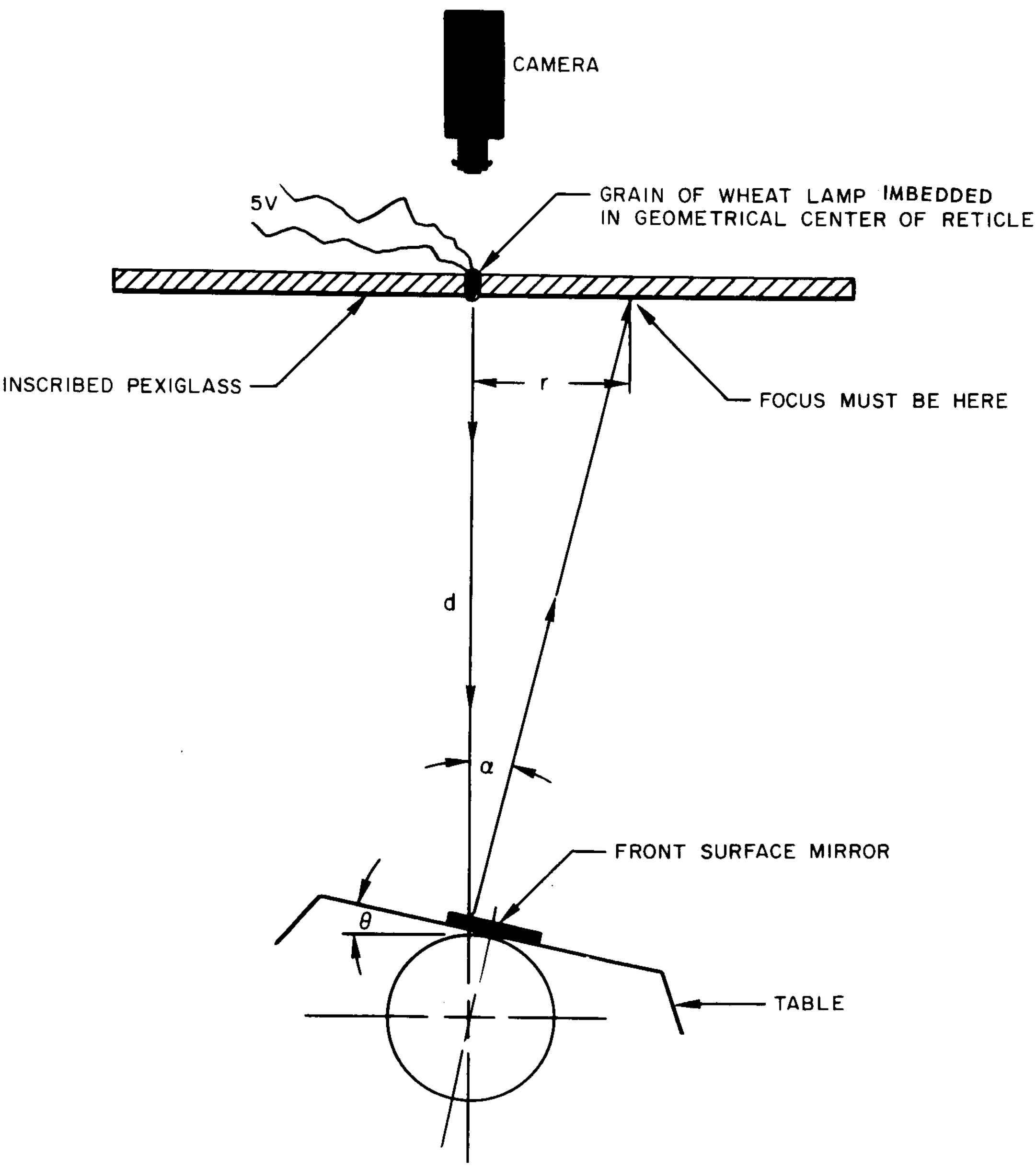
As a result of this, it was decided to try additional approaches.

W. G. Grenier 3-13-64
(Signature) (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Approach No. 4, Schematic



OBJECT = TO MEASURE ANGLE θ IN 0.1° INCREMENTS TO 12° INCL

Figure 1

670-18 (2/64)

W. G. Grenier (Signature) 3-13-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

PROCEDURE: GENERAL (Continued)

APPROACH NO. 5

5.1 The schematic for this approach is detailed in Figure 2, sheet 7. While at first, this approach seemed to offer the greatest promise to date, sincere difficulty was encountered in obtaining a clear photograph of both reticle and light trace. Several different sized pin holes were tried in the projector but results continued poor.

5.2 The problem was discussed with Mr. D. Zbrosky, of this Section who stated that the approach could be made to work. He stated that with properly ground lenses, mounted properly, good results should be obtainable. He further suggested that a completely new approach be taken. That approach to consist of reflecting light from a small sphere, mounted on the table, to the reticle. This too will require lenses but they will be simpler to locate, within the system.

W. G. Grenier 3-18-64
(Signature) (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Approach No. 5, Schematic

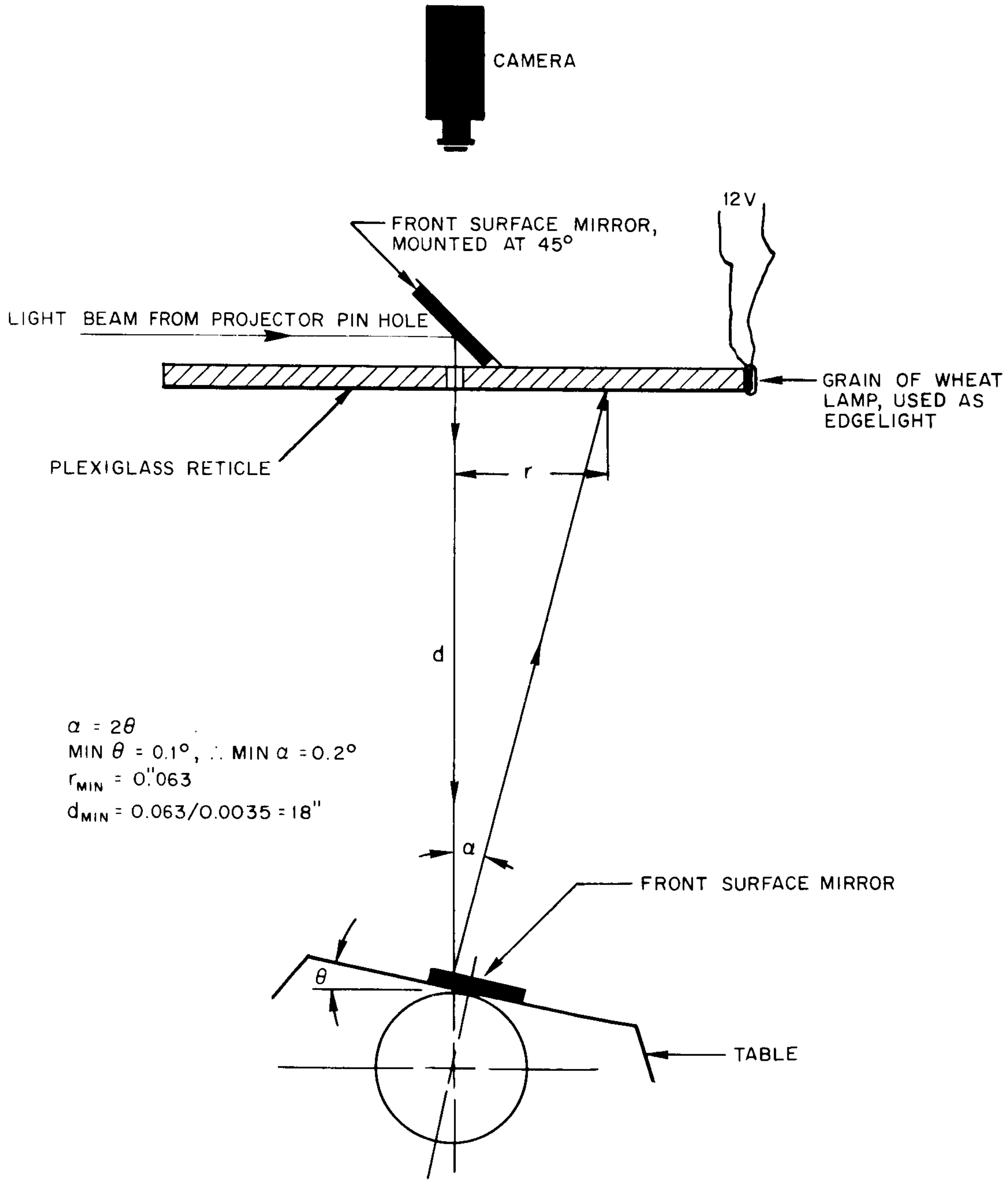


Figure 2

670-18 (2/64)

W. G. Grenier (Signature) 3-16-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

PROCEDURE: GENERAL (Continued)

APPROACH NO. 6

6.1 The schematic for this approach is given in Figure 3, sheet 9. A good trace was obtained on Polaroid film, showing both the ball point movement and the reticle design. For test purposes, the variable condenser lens from the photographic enlarger was used as a field lens. The magnifying and focusing lens, was a standard photographic enlarger lens.

6.2 Due to the success, a check was made as to the availability of components for this system. It was readily apparent that much difficulty was to be anticipated in locating a 15 inch diameter field lens. Further the weight of so large a glass lens would introduce serious mounting difficulties in other than a laboratory arrangement. It was therefore decided to change the system, such that more readily available components could be utilized.

W. G. Grenier (Signature) 3-25-64 (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Approach No. 6, Schematic

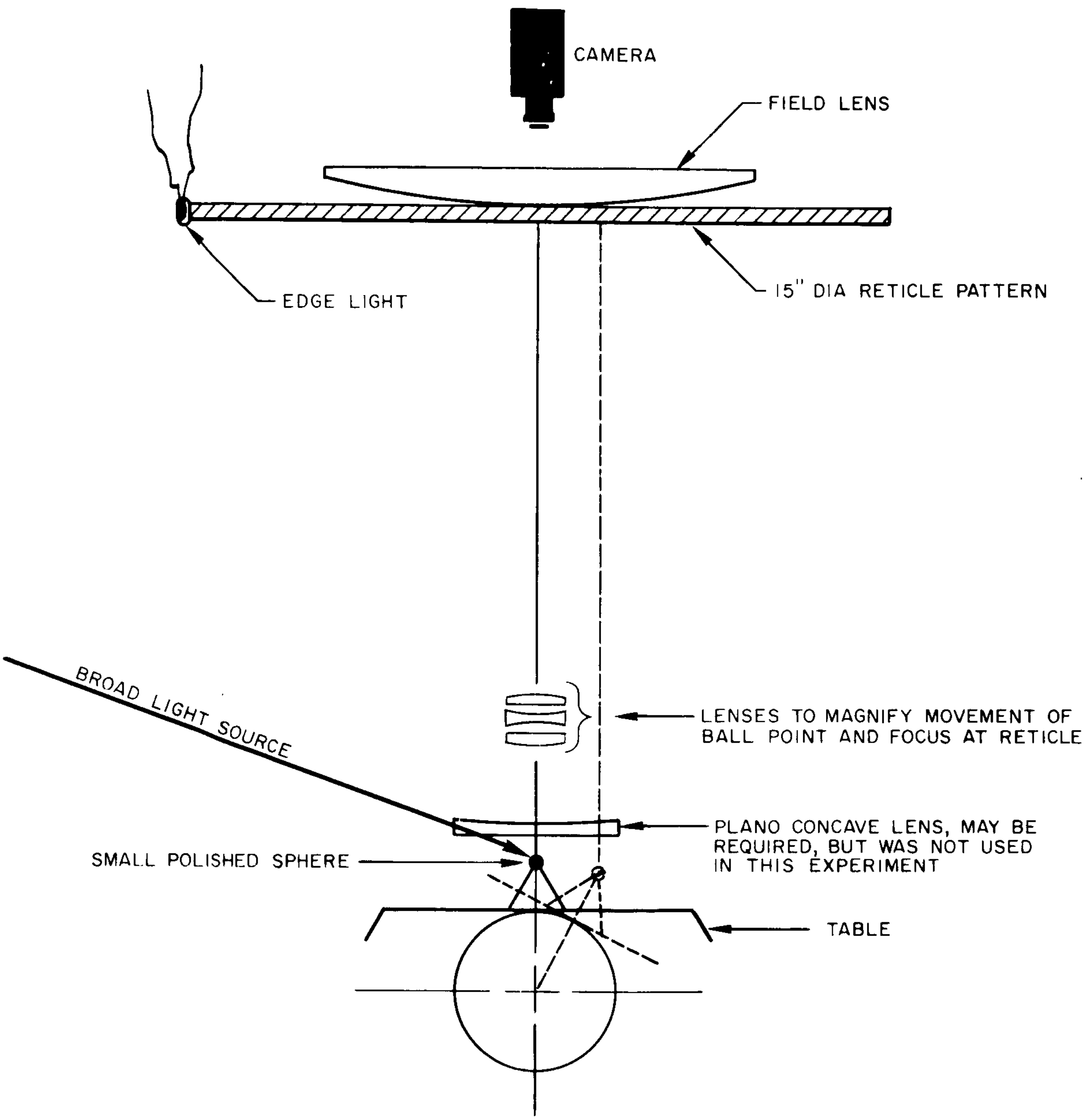


Figure 3

670-18 (2/64)

W. G. Grenier (Signature) 3-23-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

PROCEDURE: GENERAL (Continued)

APPROACH NO. 7

7.1 This approach is very similar to Approach No. 6. An edge lighted reticle will still be used, but the camera will be looking only at an image of it. This will require a field lens of only three inches in diameter.

7.2 The test setup for this approach is shown in Figure 4, sheet 11. The system produced an excellent trace with good definition of the reticle pattern. This is shown in Figure 5, sheet 12. While producing good results the system is still considered too cumbersome to be practical. Therefore an endeavor was made to refine it.

W. G. Grenier 3-25-64
(Signature) (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Test Arrangement in Approach No. 7

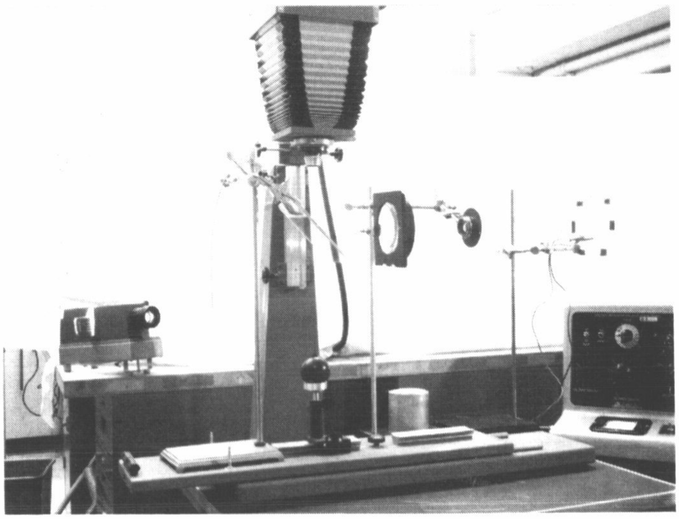


Figure 4–Test Arrangement in Approach No. 7.

Shows projector as light source, Ball point on end of wire mounted to spheroid, Beam splitter with camera mounted directly over it, condenser lens from enlarger acting as field lens, enlarger magnifying lens acting as reticle imaging lens, side lighted reticle and power source for grain of wheat lamps mounted on edges of reticle.

670-18 (2/64)

W. G. Grenier (Signature) 3-25-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Results of Approach No. 7

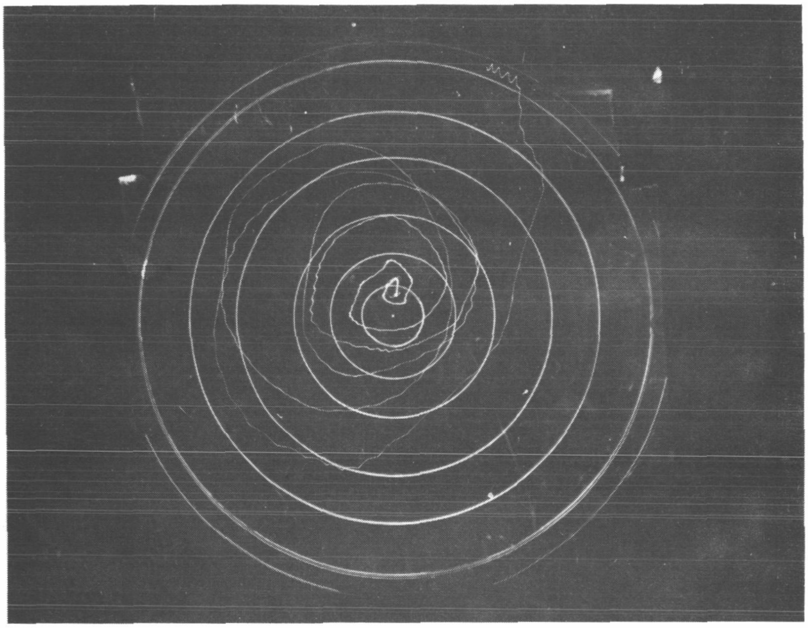


Figure 5

Photograph of trace and reticle resulting from test arrangement shown in Figure 4, on the preceding page. The spheroid was moved by hand to create the above effect.

670-18 (2/64)

W. G. Grenier (Signature) 3-25-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

PROCEDURE: GENERAL (Continued)

APPROACH NO. 8

8.1 This is an approach to reduce the bulk and complexity of the foregoing system. The reticle will be reproduced from a large master, viewed directly by the camera. It will be essentially as shown in the schematic shown in Figure 6, sheet 14.

8.2 The actual test arrangement is shown in Figure 7, sheet 15, and the results in Figure 8, sheet 16. This type system was considered acceptable by the Originator, who had been in close contact with the work at all stages.

8.3 The finalized optical schematic is given as Figure 10, sheet 20 of this report. Reticle design is given in Figure 9, sheet 17 and calculations for the reticle pattern are given in Table 1, sheets 18 and 19 inclusive.

W. G. Grenier 4-3-64
(Signature) (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Optical System Schematic, Approach No. 8

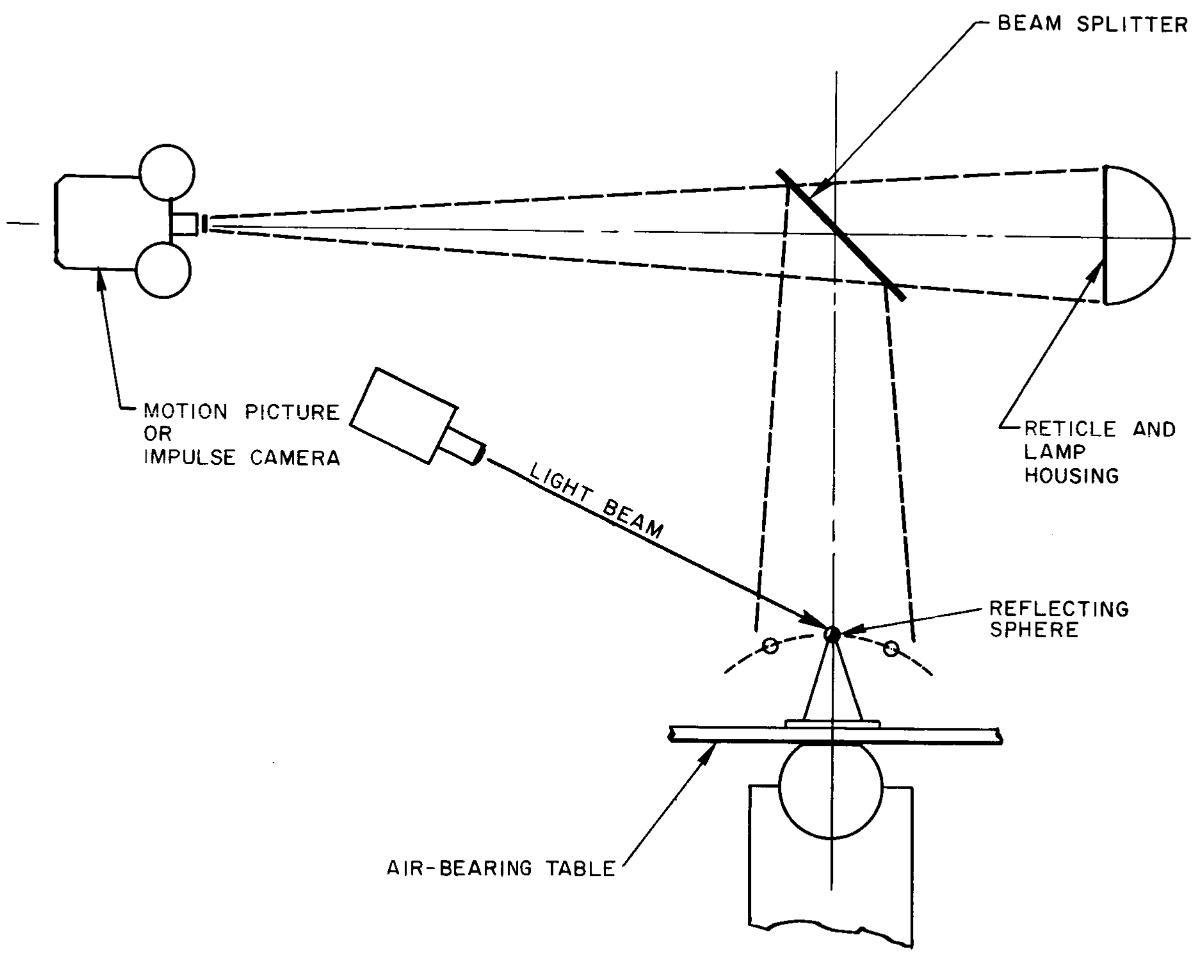


Figure 6

670-18 (2/64)

W. G. Grenier (Signature) 4-2-64 (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Laboratory Test Arrangement for Approach No. 8

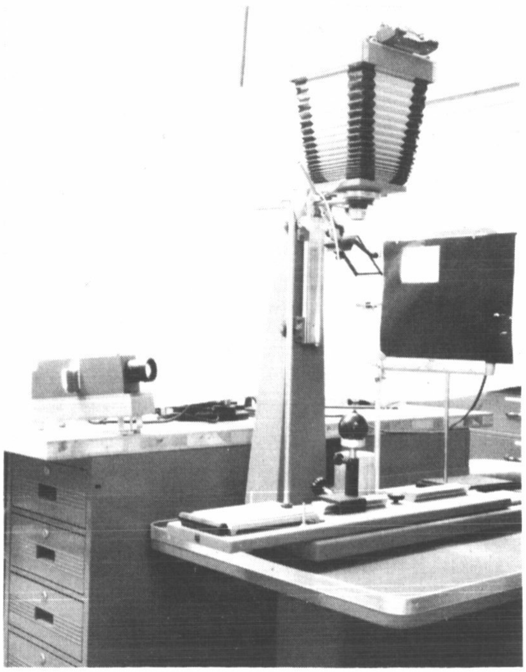


Figure 7

Shows physical arrangement of components outlined in schematic of Figure 6.

Note that the Field lens and the magnifying focusing lens have been eliminated. The trace and reticle definition are still good, as is shown in Figure 8.

670-18 (2/64)

W. G. Grenier (Signature) 3-27-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Results of Approach No. 8

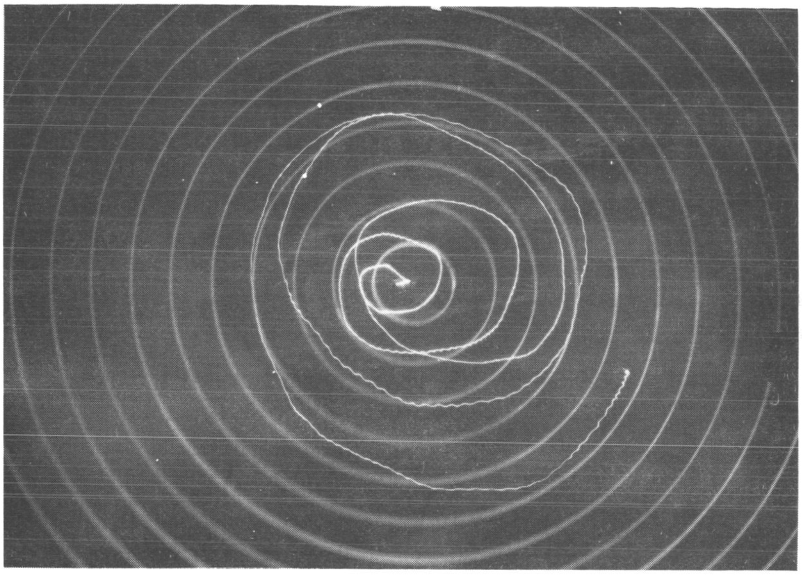


Figure 8

Photograph of trace and reticle, resulting from test arrangement described in Figures 6 and 7. The uneveness of the trace is because the sphereoid to which the ball point was attached was moved by hand.

A glass, metallographic plate, plate negative was used for the reticle.

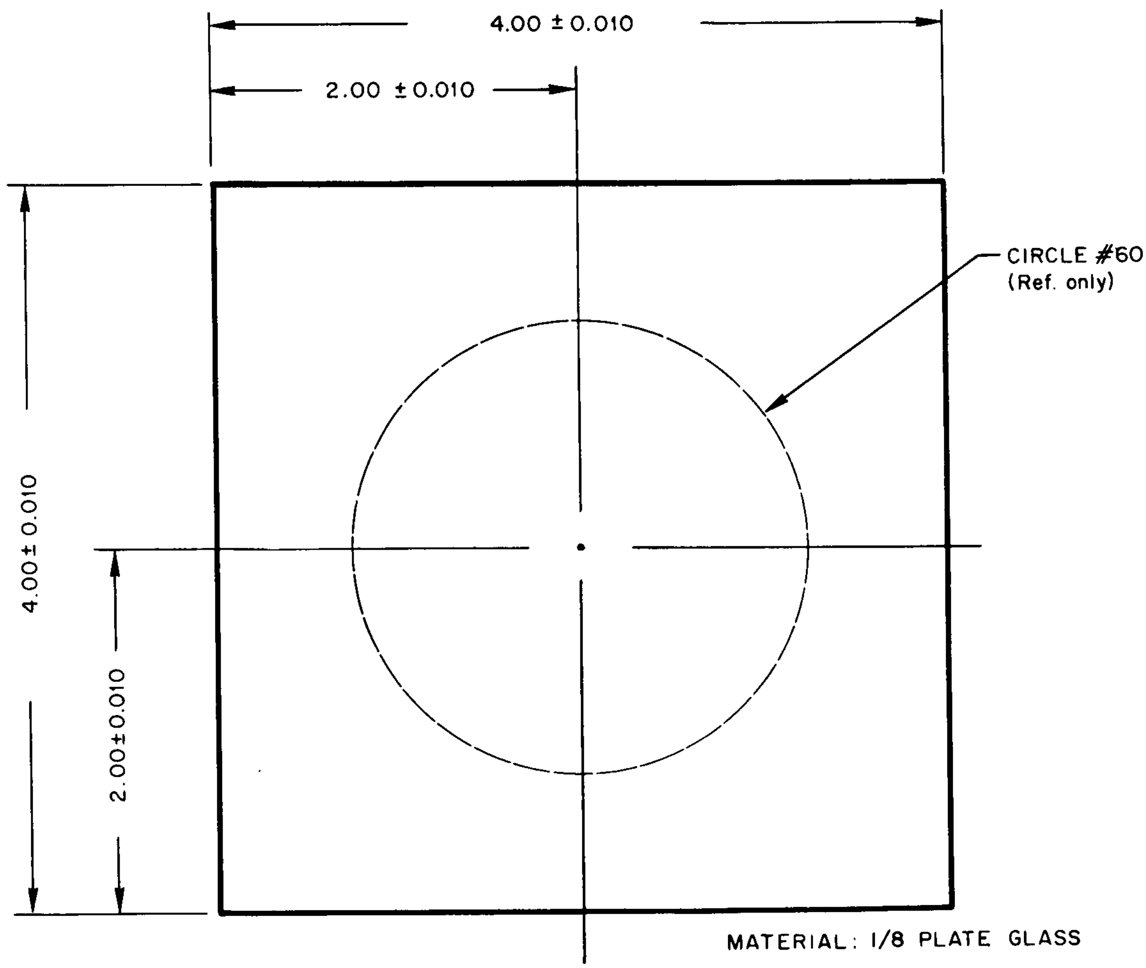
670-18 (2/64)

W. G. Grenier (Signature) 3-27-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA: Reticle Design Layout



NOTE: PIN HOLE IN CENTER TO BE 0.0005 ± 0.0002,
TO BE USED AS REFERENCE FOR CONCENTRIC CIRCLES.
RETICLE TO BE OPAQUE EXCEPT FOR SCRIBED LINES WHICH
SHALL BE CLEAR. LINE WIDTH TO BE 0.0010 ± 0.0003.
ALL DIMENSIONS IN INCHES

Figure 9

670-18 (2/64)

W. G. Grenier (Signature) 4-2-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

DATA:

Table 1
CALCULATIONS FOR RETICLE DESIGN

CIRCLE NO.	FROM FIG. 1 α IN °	SINE α	CIRC. RAD. INS.	CIRC. DIA. INS.		CIRCLE NO.	FROM FIG. 1 α IN °	SINE α	CIRC. RAD. INS.	CIRC. DIA. INS.
1	0°12'	.00349	.02094	.04188		21	4°12'	.07324	.43944	.87888
2	0°24'	.00698	.04188	.08376		22	4°24'	.07672	.46032	.92064
3	0°36'	.01047	.06282	.12564		23	4°36'	.08020	.48120	.96240
4	0°48'	.01396	.08376	.16752		24	4°48'	.08368	.50208	1.00416
5	1°0'	.01745	.10470	.20940		25	5°0'	.08715	.52290	1.04580
6	1°12'	.02094	.12564	.25128		26	5°12'	.09063	.54378	1.08756
7	1°24'	.02443	.14658	.20316		27	5°24'	.09411	.56466	1.12932
8	1°36'	.02792	.16752	.33504		28	5°36'	.09527	.57162	1.14324
9	1°48'	.03141	.18846	.37692		29	5°48'	.10106	.60636	1.21272
10	2°0'	.03490	.20940	.41880		30	6°0'	.10453	.62718	1.25436
11	2°12'	.03839	.23034	.46068		31	6°12'	.10800	.64800	1.29600
12	2°24'	.04187	.25122	.50244		32	6°24'	.11147	.66882	1.33764
13	2°36'	.04536	.27216	.54432		33	6°36'	.11407	.68442	1.36884
14	2°48'	.04885	.29310	.58620		34	6°48'	.11840	.71040	1.42080
15	3°0'	.05234	.31404	.62808		35	7°0'	.12187	.73122	1.46244
16	3°12'	.05582	.33492	.66984		36	7°12'	.12533	.75198	1.50396
17	3°24'	.05931	.35586	.71172		37	7°24'	.12879	.77274	1.54548
18	3°36'	.06279	.37674	.75348		38	7°36'	.13226	.79356	1.58712
19	3°48'	.06627	.39762	.79524		39	7°48'	.13571	.81426	1.62852
20	4°0'	.06976	.41856	.83712		40	8°0'	.13917	.83502	1.67004

670-19 (2/64)

W. G. Grenier (Signature) 3-31-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
F. Federline	Spin Control	673Y19-03	1200-62

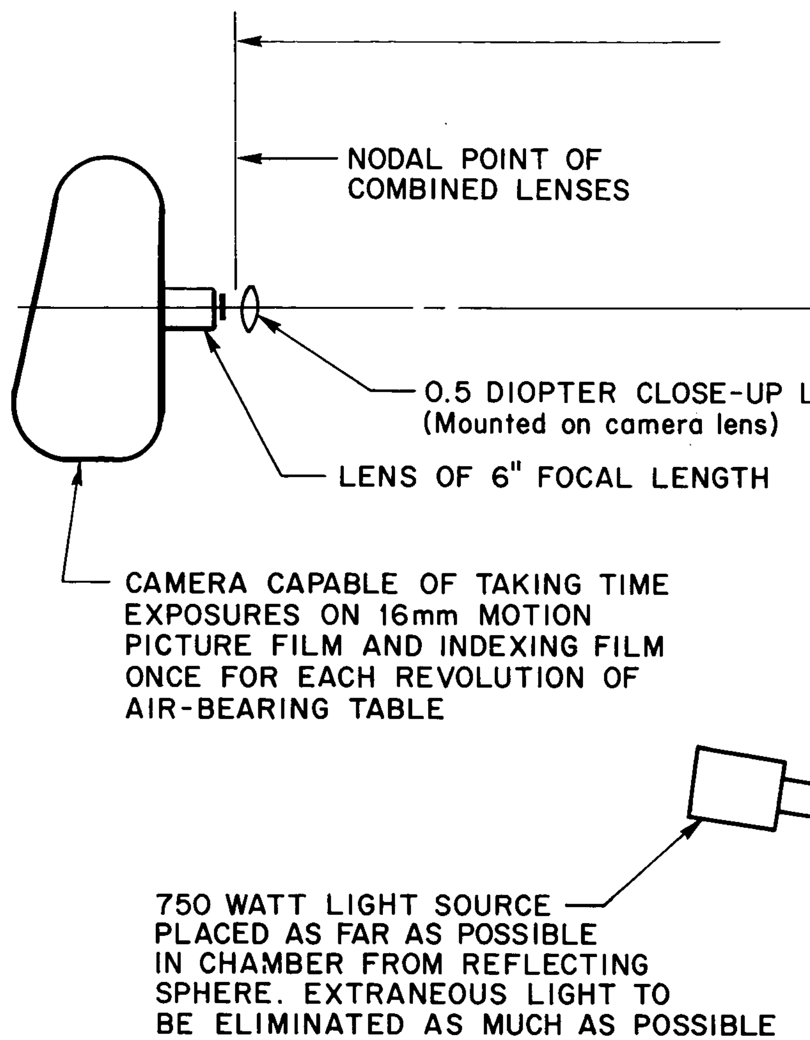
DATA:

Table 1
CALCULATIONS FOR RETICLE (Continued)

CIRCLE NO.	FROM FIG. 1 α IN °	SINE α	CIRC. RAD. INS.	CIRC. DIA. INS.		CIRCLE NO.	FROM FIG. 1 α IN °	SINE α	CIRC. RAD. INS.	CIRC. DIA. INS.
41	8°12'	.14263	.85578	1.71156		61	12°12'	.21132	1.26792	2.53584
42	8°24'	.14608	.87648	1.75296						
43	8°36'	.14953	.89718	1.79436						
44	8°48'	.15298	.91788	1.83576						
45	9°0'	.15643	.93858	1.87716						
46	9°12'	.15988	.95928	1.91856						
47	9°24'	.16333	.97998	1.95996						
48	9°36'	.16677	1.00062	2.00124						
49	9°48'	.17021	1.02126	2.04252						
50	10°0'	.17365	1.04190	2.08380						
51	10°12'	.17708	1.06248	2.12496						
52	10°24'	.18052	1.08312	2.16624						
53	10°36'	.18395	1.10370	2.20740						
54	10°48'	.18738	1.12428	2.24856						
55	11°0'	.19081	1.14486	2.28972						
56	11°12'	.19395	1.16370	2.32740						
57	11°24'	.19766	1.18596	2.37192						
58	11°36'	.20108	1.20648	2.41296						
59	11°48'	.20450	1.22700	2.45400						
60	12°0'	.20791	1.24746	2.49492						

670-19 (2/64)

W. G. Grenier (Signature) 3-31-64 (Date)


NODAL POINT OF COMBINED LENSES
0.5 DIOPTER CLOSE-UP L
(Mounted on camera lens)
LENS OF 6" FOCAL LENGTH
CAMERA CAPABLE OF TAKING TIME EXPOSURES ON 16mm MOTION PICTURE FILM AND INDEXING FILM ONCE FOR EACH REVOLUTION OF AIR-BEARING TABLE
750 WATT LIGHT SOURCE PLACED AS FAR AS POSSIBLE IN CHAMBER FROM REFLECTING SPHERE. EXTRANEOUS LIGHT TO BE ELIMINATED AS MUCH AS POSSIBLE

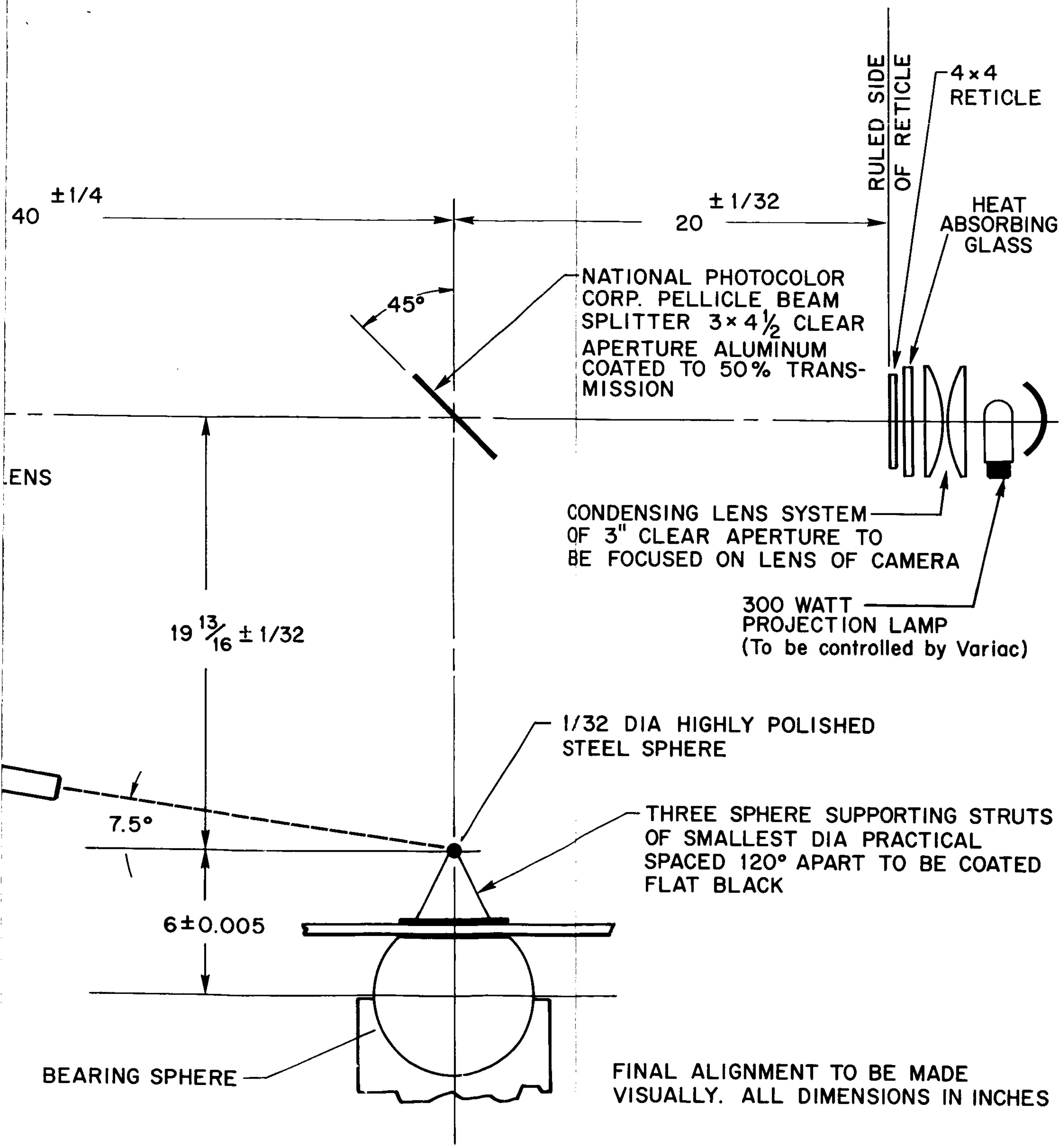


Figure 10–Schematic of Optical System Capable of Reading Coning Angle of Air Bearing Table up to 12° From Vertical.

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
P. A. Studer	11	S-126	ATD	673Y19-05	1200-64

DATE IN	DATE COMPLETED	PERFORMED BY
4-24-64	6-25-64	W. G. Grenier

NAME OF TEST

Microscopic Examination of Miniature Lamp Filaments

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Develop technique for sectioning miniature lamps and subject filaments to microscopic examination.

Two new No. 3 lamps, one new No. 9 lamp, and several burned out No. 3 lamps were tested.

EQUIPMENT INVOLVED:

Equipment normal to a well equipped Metallurgical Laboratory including, Buehler Powermet Press, DMETR bench metallurgical microscope, three wheel polishing table equipped with Automet attachments, and a B & L Research Metallograph.

RESULTS:

1. Filament wire and coil diameters are given in Table 1, sheets 4 and 5.

2. The filament material can be viewed in the microscope by visual means, but it is impractical to prepare photomicrographs, as explained on sheets 6-8 incl.

3. Visual observation, showed the filaments of both the No. 3 lamps and the No. 9 lamps to have a very fine grain structure.

W. G. Grenier (Signature) 6-25-65 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
P. A. Studer	ATD	673Y19-05	1200-64

PROCEDURE: ISOLATING LAMP FILAMENT

Employing the wet belt surfacer, equipped with a 120 grit silicon carbide belt, carefully grind away a portion of the glass covering. Hold the miniature lamp in tweezers for this operation. In the first try, the tips of the tweezers were partially ground away and when the break through of the glass casing occurred, the said glass shattered. Following several additional attempts a method was arrived at which is fairly reliable.

The miniature lamp is held carefully between thumb and forefinger of one hand. The tip of the lamp is abraded, dry, against a 240 grit, or finer, silicon carbide abrasive paper. The abrading is continued until with the aid of a hand magnifier it can be ascertained that one is very close to the filament. The lead wires are then grasped firmly between the thumb and finger of one hand. The lamp is laid carefully on the abrasive paper, such that the filament is aligned generally in a plane parallel to the abrasive plane. Using one finger of the other hand, apply a light pressure to the lamp, while pulling it over the paper. Repeat the strokes, as necessary, until the filament is adequately exposed for microscopic examination without interference from the glass shell. This procedure exposes the filament for measurement and surface examination only.

Following measurement of the filament wire diameters and coil diameters an attempt was made to mount the lamps in lucite. Each specimen so mounted was carefully hand polished in an endeavor to reveal the filament cross section to view.

W. G. Grenier (Signature) 6-22-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
P. A. Studer	ATD	673Y19-05	1200-64

PROCEDURE: FILAMENT AND COIL DIAMETER MEASUREMENTS

Having isolated the lamp filament from it's protective glass shell, the diameters of the coil and filament can now be determined. The Bausch & Lomb DMETR Metallurgical Microscope shall be used in conjunction with the Filar Micrometer Eyepiece. To obtain the maximum depth of focus and practical resolution, the 20X, 0.40 N.A. objective is to be used. This combination was previously calibrated, on this instrument, when it was determined that one Filar unit is equal to 17.5×10^{-6} inches. It was further noted that the filar hair in the eyepiece was equal to two filar units in thickness. The results of the above measurements are given in Table 1 of the data.

NOTE: All lamps examined are purportedly products of Los Angeles Miniature Products, Inc.

W. G. Grenier (Signature) 6-22-64 (Date)

NO. 11
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
P. A. Studer	ATD	673Y19-05	1200-64

DATA:

Table 1
FILAMENT WIRE & COIL, DIAMETER MEASUREMENTS

LAMP SIZE NO.		CONDITION		FILAMENT WIRE DIAMETER F.U.	FILAMENT WIRE DIAMETER INS. × 10^{-4}		COIL DIAMETER F.U.	COIL DIAMETER INS. × 10^{-4}		
3		Burned Out		14	2.45		111	19.4		
				12	2.10		105	18.4		
				14	2.45		109	19.1		
				13	2.27		107	18.7		
3			AVERAGE		2.32		–	18.9		
3		New		12	2.10		107	18.7		
				13	2.27		105	18.4		
				12	2.10		106	18.6		
				11	1.92		100	17.5		
				9	1.58		100	17.5		
3			AVERAGE		1.99		–	18.1		
3		New		10	1.75		110	19.2		
				13	2.27		104	18.2		
				9	1.58		106	18.6		
				11	1.92		105	18.4		
3			AVERAGE		1.88		–	18.6		

670-19 (2/64)

W. G. Grenier (Signature) 6-23-64 (Date)

NO. II
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
P. A. Studer	ATD	673Y19-05	1200-64

DATA:

Table 1
FILAMENT WIRE & COIL, DIAMETER MEASUREMENTS (Continued)

LAMP SIZE NO.		CONDITION		FILAMENT WIRE DIAMETER			COIL DIAMETER			
				F.U.	INS. × 10^{-4}		F.U.	INS. × 10^{-4}		
9		New		35	6.12		330	57.7		
				36	6.30		328	57.4		
				31	5.43		328	57.4		
				35	6.12		325	56.8		
				32	5.60		327	57.2		
9			AVERAGE		5.91		–	57.3		

670-19 (2/64)

W. G. Grenier (Signature) 6-23-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
P. A. Studer	ATD	673Y19-05	1200-64

PROCEDURE: FILAMENT METALLOGRAPHY

Those lamps whose filaments had been exposed, as described on sheet 2 of this report, were mounted in lucite. The Buehler Powermet Press was used to form the mounts. In each case, the operator was careful not to apply full pressure to the mold until the maximum mold temperature of 150°C, had been held for at least five minutes. This was done in an effort to minimize distortion of the filament wire, since at that temperature the lucite would be expected to be most fluid. In spite of this precaustion each filament was bent out of its original form and location. Lucite was chosen as the mounting medium, rather than a harder one, due to the recognized difficulty in locating the very small filament wires. This difficulty would have been compounded had a dense opaque mounting medium been used.

Each specimen was ground on the wet belt surfacer until, by viewing with a hand magnifier, some part of a filament was ascertained to be near the surface. Rough polishing was continued on the Buehler Handimet hand polisher until some portion of each filament was polished. Polishing in this manner was conducted with frequent microscopic checks as to polishing effect.

Fine polishing was accomplished by means of the three wheel, slow speed polishing table, equipped with Automet attachments for each wheel. Mechanical polishing was as follows:

W. G. Grenier (Signature) 6-25-64 (Date)

670-15

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
P. A. Studer	ATD	673Y19-05	1200-64

PROCEDURE: FILAMENT METALLOGRAPHY (Continued)

1. 600 grit silicon carbide paper, with copious quantities of water, 40 lbs. pressure–2-1/2 minutes.
2. 15 micron sized diamond on Buehler Texmet cloth, 40 lbs pressure–1 minute.
3. 6 micron sized diamond on Buehler Texmet cloth, 50 lbs pressure–2 minutes.
4. Fishers Gamal–Grade B, <0.1 micron sized abrasive, suspended in deionized water on Buehler Texmet cloth, 50 lbs. pressure–2 minutes.

Subsequent to the final mechanical polish each specimen was chemically polished prior to etching. Chemical polishing is accomplished by swabbing vigorously, for a period of time up to four minutes with the following solution:

Lactic Acid – 30 ml

Nitric Acid – 10 ml

H F – 5 ml

This effected an excellent strain free polish.

Each specimen was then etched, by immersion in the following:

$K_3 Fe(CN)_6$ – 12 gm

Na OH – 1 gm

H_2O (distilled) – 100 ml

W. G. Grenier 6-25-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
P. A. Studer	ATD	673Y19-05	1200-64

PROCEDURE: FILAMENT METALLOGRAPHY (Continued)

This procedure revealed a very fine grain structure in the filaments of both numbered lamps. However the specimens were too minute and rounded to obtain usable photomicrographs.

W. G. Grenier 6-25-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
P. A. Studer	ATD	673Y19-05	1200-64

CONCLUSIONS:

The dimensions reported herein verify earlier figures obtained from microscopic examination through the glass envelope and those derived from electrical measurements.

The fact that the filament diameter of the "burned out" lamp (#3) was not reduced in size indicates that evaporation rate phenomena was not the determining factor in "burn out."

Apparently metallographic examination is impractical for use in obtaining significant data for quality control on miniature lamp filaments, due to the small cross sectional areas.

P. A. Studer (Signature) 6-29-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
C. E. Vest	11	S-120	ATD	673Y03-14	1200-49

DATE IN	DATE COMPLETED	PERFORMED BY
9-11-63	Interim	W. G. Grenier and J. L. Wall

NAME OF TEST

Codeposition Study

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Determination of feasibility of codepositing Ni and MoS_2, as a resevoir technique for supplying a lubricant in space applications.

EQUIPMENT INVOLVED:

Well equipped Metallurgical Laboratory.

RESULTS:

1. Wetting of the MoS_2 powder was accomplished using Triton X-100 in the proportion of 0.13 ml Triton per gram of powder per liter electrolyte.

2. Photomacrographs: See Figures 2-6 inclusive, pages 15-17 of the data.

3. Photomicrographs: See Figures 7-12 inclusive, pages 18-21 of the data.

In accordance with verbal instructions issued on 5-1-64 no further work will be done on this project.

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: GENERAL

I. APPROACH

It was desired that a technique be developed for the electro-codeposition of micron sized Mo S_2 particles and various basis metals. As complete knowledge of the mechanisms involved in this type of electrodeposition is not available, an empirical approach was taken. Nickel plating techniques are well known and nickel plating electrodes are readily available. Therefore it was determined to endeavor to codeposit nickel and Mo S_2 particles, as a preliminary step. This step is to be followed by more sophisticated steps until the requisite techniques are established.

The Originator suggested that a preliminary feasibility investigation be undertaken. This report contains the procedures followed and the data obtained in the feasibility study.

II. Mo S_2 POWDER, DEFINED

For purposes of this report, Mo S_2 powder will mean, the particular micro sized Mo S powder herein described. That Mo S_2 powder, used in every instance, was purchased under the Alpha Molykote trade name of "Molykote Microsize Powder." According to the Alpha Molykote literature, that powder has the following physical characteristics:

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: GENERAL

II. MoS_2 POWDER, DEFINED (Continued)

Particles from 0 – 1/2 micron = 43% of the total powder

Particles from 1/2 – 1 micron = 20% of the total powder

Particles from 1 – 2 micron = 23% of the total powder

Particles from 2 – 3 micron = 10% of the total powder

Particles from 3 – 4 micron = 2% of the total powder

Particles from 4 – 5 micron = 0.8% of the total powder

Particles from 5 – 8 micron = 1.2% of the total powder

III. PREPLATE CLEANING

Preplate cleaning was identical for all samples tested. This consisted of cleaning ultrasonically, for two minutes each in Propanol, followed by Trichloroethane, followed by Benzene. Upon removal from the ultrasonic cleaner the samples were retained in Benzene until ready for activation or plating.

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: GENERAL

IV. ACTIVATION

Not all samples were subjected to an activation treatment. The only activation solution used was 20 v/o H_2 So_4 in distilled water. For purposes of this report, it is to be understood that activation was not employed, unless specifically stated.

V. PLATING BATH

With the exception of the final plating bath used in this study, all plating baths consisted of the following composition:

Ni Cl_2	– 25 gms per liter
H Cl	– 12 v/o
H_2O	– Distilled
Mo S_2 powder	– 1 gram per liter

All plating was done at room temperature with smooth nickel electrodes supplied by the Fabrication Division.

W. G. Grenier 4-9-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: GENERAL

V. PLATING BATH (Continued)

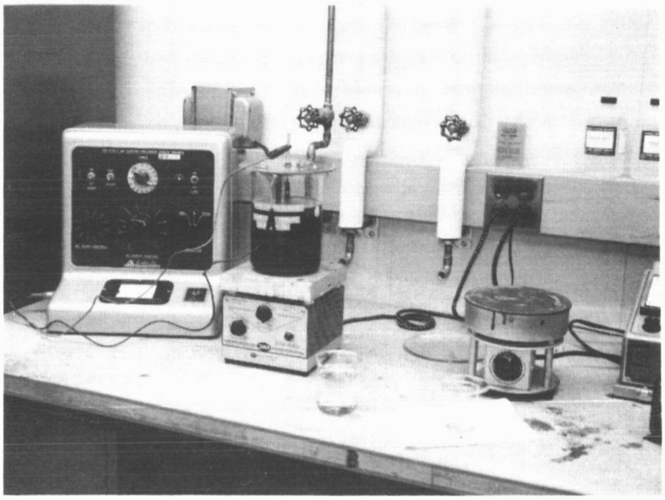


Figure 1–Co Deposition Equipment – Set Up.

From left to right: D.C. power source, magnetic stirrer, plating bath in beaker, small beaker of distilled water, covered beaker containing 20 v/o H_2 So_4 solution and a hot plate. Almost out of the photograph is a pH meter used to determine the acidity of the plating bath. In some early tests the pictured power source was replaced with dry batteries.

W. G. Grenier 4-9-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: EXPERIMENTAL WETTING OF MoS_2 MICRO SIZED POWDER

A prerequisite to codepositing the MoS_2 particles, with any basis metal, was dispersion of a quantity of MoS_2 powder in the electrolyte. Therefore it was necessary to wet the powder, such that a uniform dispersion could be maintained with mechanical agitation. Uniform mechanical agitation was assured by use of the Tempco magnetic stirrer shown in Figure 1. Several approaches to wetting the MoS_2 powder were undertaken and evaluated, as follows:

1. Wetting with Ethanol

   1.1 Mixed 1 gram MoS_2 powder with 20 ml ethanol

   1.2 Poured mixture into 750 ml electrolyte

   1.3 It appeared that approximately 1/2 of the powder dispersed into the electrolyte, while the other 1/2 floated on the surface.

2. Wetting with Benzene

   2.1 Prepared 1 gm MoS_2 powder and 50 ml Benzene.

   2.2 The Benzene appeared to wet the MoS_2 powder but did not separate when poured into the electrolyte.

   2.3 The electrolyte was heated in an effort to drive off the Benzene.

   2.4 In spite of mechanical agitation and heat, the MoS_2 powder floated to the surface.

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: EXPERIMENTAL WETTING (Continued)

2.5 Sprayed the Mo S_2 powder, floating on the surface with ethanol. Some of the powder dispersed in the electrolyte under agitation.

2.6 Skimmed the surface of the electrolyte removing that powder which was floating. A small proportion of the powder appeared to be in suspension.

3. Wetting with Ethanol–Second approach

3.1 Using a small beaker and stirring constantly, Mo S_2 powder was added to 5 ml of Ethanol until a thick creamy paste was formed.

3.2 Electrolyte solution was added to the paste, by means of a dropping pipette, a few drops at a time. Stirring continued constant until the blended mixture appeared sufficiently fluid to add to the main container of electrolyte.

3.3 The fluid was poured into the main container of electrolyte and agitated.

3.4 An apparent major portion of the Mo S_2 powder separated and floated on the surface of the electrolyte.

4. Wetting with a Detergent, i.e., Fisher's Sparkleen

4.1 To 50 ml of electrolyte add random amount of Mo S_2 powder. Note that powder floats on the surface and seems to cling to the sides of the beaker.

4.2 To 50 ml of tap water, add 10 gms of Sparkleen detergent powder. Stir until all detergent is in solution.

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: EXPERIMENTAL WETTING (Continued)

4.3 Using a dropping pipette, drops of detergent solution were added to the beaker containing electrolyte and Mo S_2 powder.

4.4 The Mo S_2 powder appeared to immediately disperse into the electrolyte.

5. Wetting with Sparkleen—Further Tests

5.1 Several drops of that detergent solution from 4.2 were placed on the sides of the electrolyte container from 3.3. That container was in the condition described in 3.4 above. As the detergent solution reached the electrolyte surface, some of the Mo S_2 powder, on the surface and sides of the container, was instantly dispersed.

5.2 The contents of the beakers in 4.2 and 4.4 were added to the main electrolyte of 3.3, with agitation from the magnetic stirrer. All Mo S_2 powder dispersed in the electrolyte.

5.3 With discontinuance of agitation the Mo S_2 powder settled to the bottom of the electrolyte. There was no skim on the surface.

5.4 The foregoing indicates that good wetting was achieved with the detergent. However, this particular detergent is a strong alkali. Therefore a more nearly neutral wetting agent would be desirable, such as Triton X-100.

670–15

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: EXPERIMENTAL WETTING (Continued)

6. Wetting with Triton X-100

6.1 To 20 ml of tap water add 5 ml of 25 v/o Triton X-100. This is a 6.25 v/o Triton X-100 solution.

6.2 An unmeasured amount of Mo S_2 powder was added to 6.1. It dispersed in the solution on contact.

6.3 The contents of 6.2 were poured into a beaker containing some Ni Plating electrolyte with no agitation. The dispersion appeared excellent.

6.4 An effort was made to determine the minimum quantity of Triton X-100 required to disperse 1 gm of Mo S_2 powder in 1 liter of electrolyte. After several trials, it was learned that it required 0.13 ml Triton X-100 concentrate per liter of electrolyte to disperse 1 gm of Mo S_2 powder.

6.5 It was noted that as the quantity of Mo S_2 powder is increased, so is the required amount of wetting agent. No attempt at a positive correlation was made.

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: CODEPOSITION OF Ni AND Mo S MICRO SIZED PARTICLES

1. Employing two specimens of 7075 aluminum, a trial run was made using a pair of 9 volt dry batteries connected in parallel. See Article III in the General Procedures for preplate cleaning. See Experimental Wetting – paragraph 1, for procedures followed in adding the Mo S_2 powder to the electrolyte. In this trial the current was varied indiscriminately until all current available was used. The resultant coating was black and nubby. No attempt at evaluation was made at this time, nor were the specimens retained. This trial was simply part of an attempt to obtain a feel for the process.

2. A sample of stainless steel was activated as per Article IV in the Genral Procedures, for one minute at room temperature. This trial was then conducted using the same power source and plating bath as in 1 above. Regardless of current density, this sample did not coat as uniformaly as did the 7075 aluminum specimens. An additional stinless steel sample, subjected to identical preplate treatment, was plated in Ni plating solution containing no Mo S_2 powder. The resultant plate was a dull gray and at high current densities, yielded the expected nubby textured surface. At this point, the Electropolisher DC power supply was substituted for the dry batteries and was used exclusively throughout the balance of the tests. This arrangement is shown in Figure 1.

3. Another rough experiment was conducted employing a strip of 300 series stainless steel, whose approximate dimensions were 1" × 4" × 1/8". One end of the strip was

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: CODEPOSITION (Continued)

immersed in the plating bath containing the MoS_2 powder. The immersed area was estimated and a plating current density of approximately 50 milliamperes per square inch was applied. A high rate of gassing was observed at the sample throughout a one hour plating time. After one hour; the strip was removed, the ends reversed and the second end plated at the same current density for two hours. Subsequent to the two hour run; the sample was removed from the codeposition bath, rinsed in distilled water and allowed to air dry. The surface appeared black and rough. Each end of the dried strip was sectioned on the abrasive cutoff machine, mounted in glass filled epoxy and prepared for Metallographic examination. The sectioned specimens were so orientated that a transverse and longitudinal cross sectional area were observable in each mount. Each specimen was rough ground on the wet-belt surfacer and polished on the three wheel, slow speed, polishing table utilizing the Automet attachments. Final polish was achieved using; Gamal alumina on well dressed microcloth, an Automet setting of 35 pounds and a polishing time of 3 minutes. The specimens were examined at magnifications to 1500 diameters. No evidence of codeposited MoS_2 particles and Ni were discovered.

4. It was felt that a distinct possibility existed of there being too small a quantity of the Mo S powder dispersed in the codeposition bath. Therefore the series of experiments on wetting MoS_2 powders, mentioned earlier in this report, were conducted. The methods

W. G. Grenier 4-9-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: CODEPOSITION (Continued)

of experiment number 6, Wetting with Triton X-100, were used exclusively throughout the balance of this feasibility study.

5. Three samples of AISI-410 stainless steel were prepared in accordance with Articles III & IV of the General Procedures. Activation was conducted at room temperature. The average surface area of each sample was calculated to be 1.36 square inches. The plating bath used is described in Article V of the General Procedures. The three samples were coated individually at differing current densities and for differing plating time periods.

5.1 One sample was subjected to a plating current density of 100 milliamperes per square inch, for one hour with no apparent deposit. The current density was increased to 200 milliamperes per square inch and held for one hour. At the end of this time, the sample appeared coppery in color and felt slick to the touch. Rubbing with a tissue caused some gray coloration to show.

5.2 Another sample was subjected to a plating current density of 300 milliamperes per square inch, for one hour in the same plating bath. At the end of that time, the sample was rinsed in distilled water and allowed to air dry. The results are shown in Figures 2 and 3 of the data.

W. G. Grenier 4-9-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: CODEPOSITION (Continued)

5.3 The sample used in 5.2 above, was cleaned by means of abrasive papers and prepared again for coating, as originally. It was subjected to a plating current density of 400 milliamperes per square inch for one hour. At this time it appeared that the sample might have a flash coating of nickel. It was promptly replaced in the plating bath and held at a current density of 600 milliamperes per square inch for another hour. At the end of this period, it appeared somewhat polished with miscellaneous deposits in evidence, as shown in Figures 4 and 5 of the data.

5.4 The third sample was placed in the plating bath and subjected to a current density of 6290 milliamperes per square inch. It was checked at the end of one half of an hour and appeared to have a coating 0.0004 inches in thickness. The sample was replaced in the plating bath and held at the same current density for an additional 2-1/2 hours. At the end of that time the sample had a very heavy, very flaky coating. A majority of the flakes blew off readily when subjected to an air blast. However, that coating on the edges of the sample appeared to adhere tightly. See Figure 6, of the data. It was hoped that some of the color of the coating was due to Mo S_2 particles in the nickel. Therefore a Metallographic examination was conducted. No Mo S_2 particles were defined in the nickel matrix, as is shown in the photomicrographs. See Figures 7-10 inclusive, of the data.

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

PROCEDURE: CODEPOSITION (Continued)

5.5 It was observed, during the plating cycle of 5.4, that gas bubbles forming at the electrodes appeared to entrap some of the Mo S_2 powder. These bubbles and powder floated to the surface of the plating bath and remained there throughout the plating cycle. Upon cessation of current application and bath agitation, all Mo S_2 particles settled to the bottom of the plating bath.

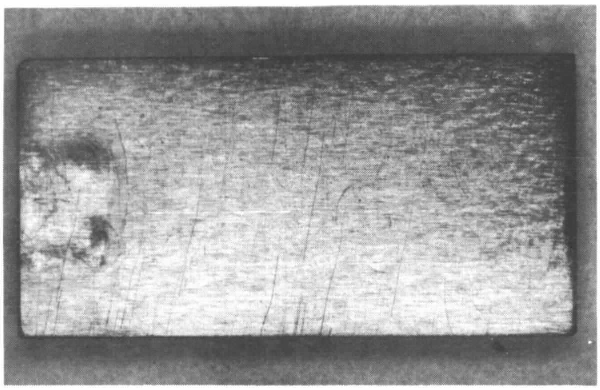
6. Another approach was tried. An unknown quantity of wetted Mo S_2 powder, believed to be 150 grams or more, was added to the foregoing plating bath. An additional unknown amount of Ni Cl_2 and H Cl was also added to the plating bath. The latter was added to counteract possible effects of the additional Triton X-100, required to wet such copius quantities of the Mo S_2 powder. Another identical AISI-410 stainless steel sample was prepared. It was subjected to a plating current density of 7000 milliamperes per square inch, for 3/4 hours. This resulted in an extremely uneven coating, as was expected, which did not have the appearance of a simple, dull nickel plate. The sample was sectioned and prepared for Metallographic examination. The examination revealed agglomerated Mo S_2 particles entrapped in the nickel matrix. See Figures 11 and 12 of the data.

W. G. Grenier 4-9-64
(Signature) (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

DATA: Photomacrographs: Magnification = 3 diameters



X

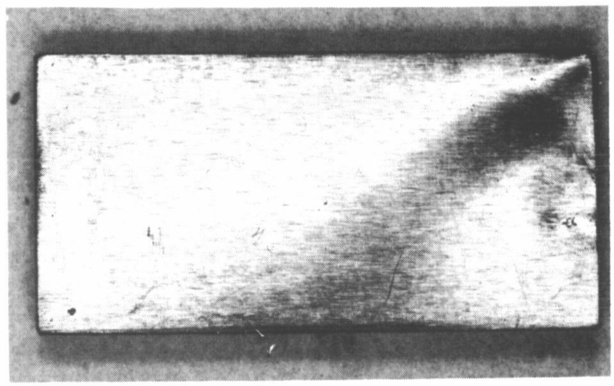


Figure 2

Figure 3

Figures 2 and 3 show two faces of a 410 S.S. sample after attempting to codeposit Mo S_2 and Ni.

Current density = 300 ma per square inch
Plating Time = 1 hour

The sample was held by means of an alligator clip at the end designated by X.

Deposit Thickness = too thin to measure

Note lack of uniformity in possible buildup.

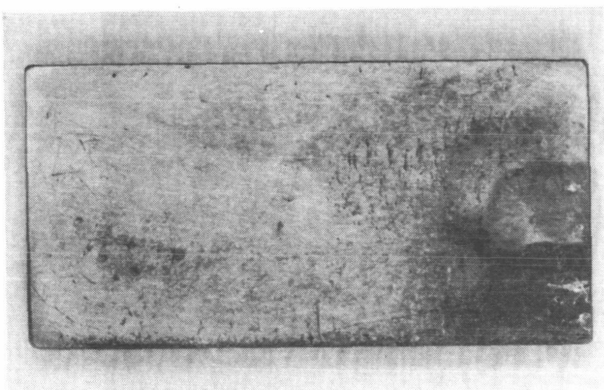
670-18 (2/64)

W. G. Grenier (Signature) 4-9-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

DATA: Photomacrographs: Magnification = 3 diameters



X

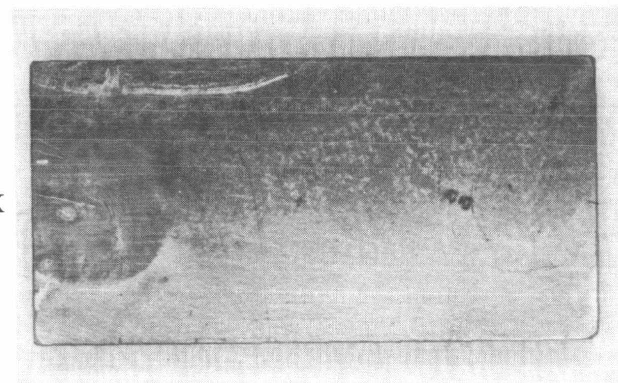


Figure 4

Figure 5

Figures 3 and 4 show two faces of a 410 S.S. sample after attempting to codeposit Mo S_2 and Ni.

Current Density = 400 ma per square inch for one hour, followed by 600 ma per square inch for one hour.

There was a marked coppery coloration at the electrode contact points designated by X.

The deposit was too thin to measure.

670-18 (2/64)

W. G. Grenier (Signature) 4-9-64 (Date)

NO. 1
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

DATA: Photomacrograph: Magnification = 3 diameters

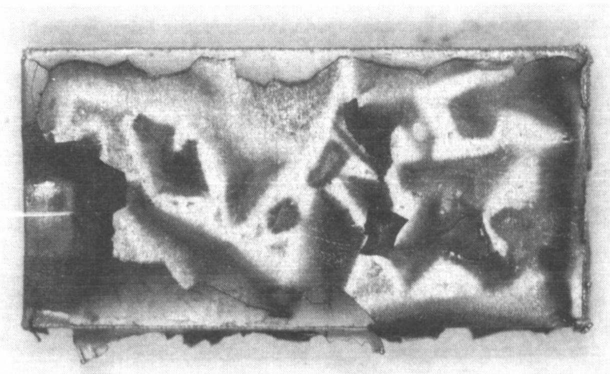


Figure 6

Same 410 stainless steel sample as that used in Figures 4 and 5. Sample was held in codeposition bath for 3 hours at a current density of 6290 milliamperes per square inch. That is 905 amperes per square foot.

Note the obvious flakiness. The plate appeared typical for a dull nickel plate at this current density. The white areas are locations where flakes of the coating appeared to adhere but blew off when subjected to an air stream. That coating shown on the top edge of the sample appeared tight. Therefore this sample was sectioned and polished for Metallographic examination, to determine possible presence of codeposited Mo S_2 particles in the nickel.

670-18 (2/64)

W. G. Grenier (Signature) 4-9-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

DATA: Photomicrograph: Magnification = 750 diameters

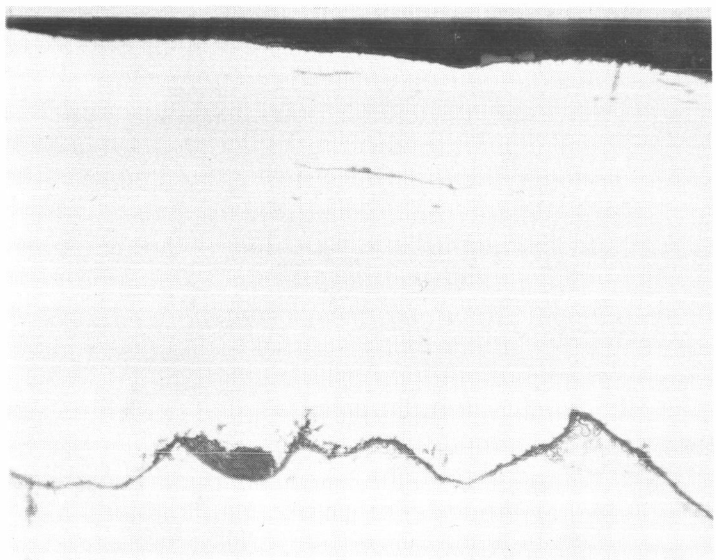


Figure 7

Shows typical, as polished, condition of specimen from sample shown in Figure 6.

No Mo S_2 particles are in evidence in the nickel plate. Some spotting from solvents and substrate corrosion products due to the activation process, are in evidence at the interface.

670-18 (2/64)

W. G. Grenier (Signature) 4-9-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

DATA: Photomicrograph: Magnification = 750 diameters

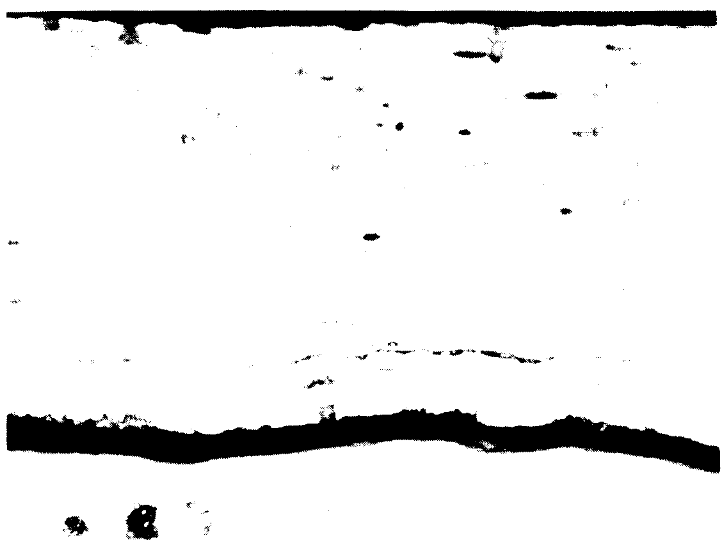


Figure 8

Typical coating on specimen from Figure 6 after light etch.

No Mo S_2 particles are apparent in the nickel plating. Discontinuities in the plate are probably caused by nickel particles and gassing at the unusually high current density.

Etch: 10% Oxalic-electrolytic, followed by Picral-H Cl immersion, followed by 50/50 H NO_3-Acetic, immersion.

670-18 (2/64)

W. G. Grenier 4-9-64
(Signature) (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

DATA: Photomicrographs: Magnification = 750 diameters

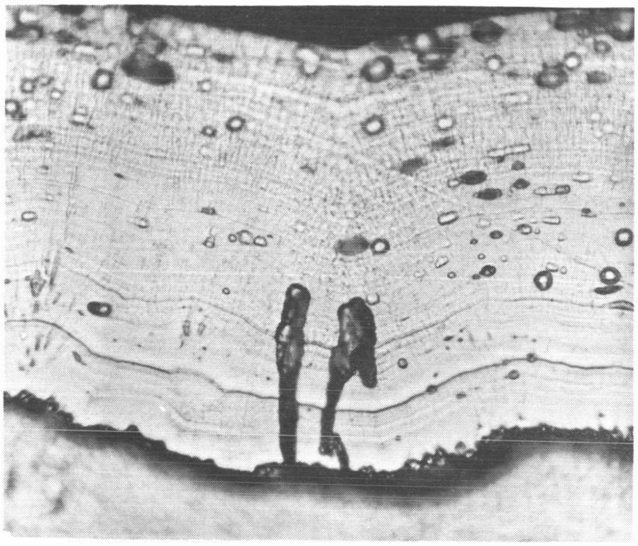


Figure 9–Bright Field Illumination

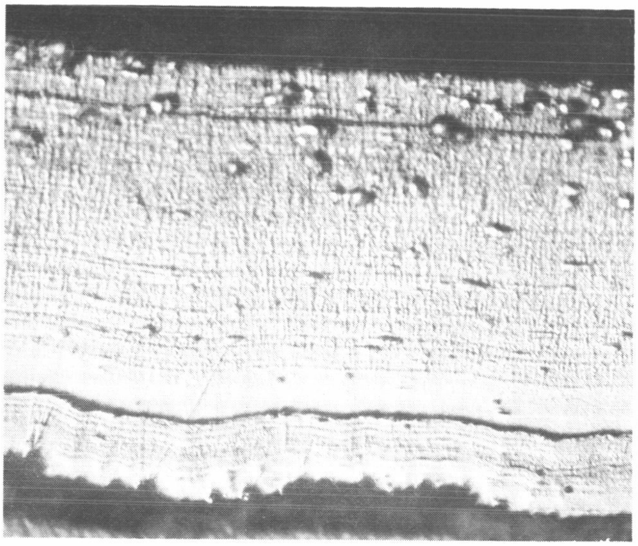


Figure 10–Oblique Illumination

Figures 9 and 10 show typical areas of the specimen shown in Figures 6-8 inclusive after repolishing and reetching to verify findings.

Etch: Picral–H Cl, for stainless substrate microstructure
$H\ NO_3$ –Acetic, for Ni plating microstructure

No $Mo\ S_2$ particles are in evidence in the nickel plating matrix. Oblique illumination, shown in Figure 10, points up the striations and embedded nickel particles typical of nickel plate at high current densities with unbagged electrodes.

670-18 (2/64)

W. G. Grenier (Signature) 4-9-64 (Date)

NO. I
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

DATA: Photomicrographs: Magnification = 250 diameters

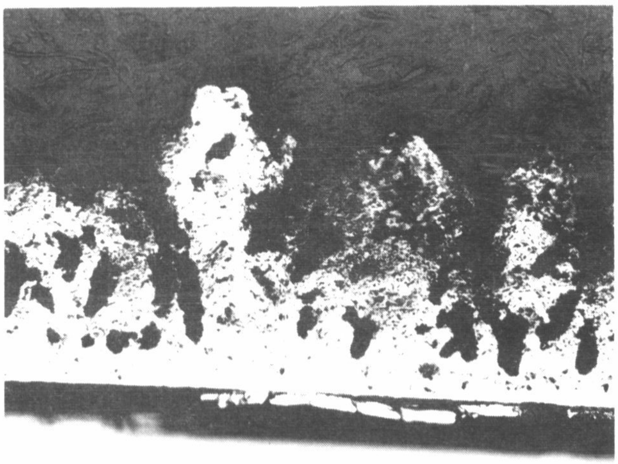


Figure 11

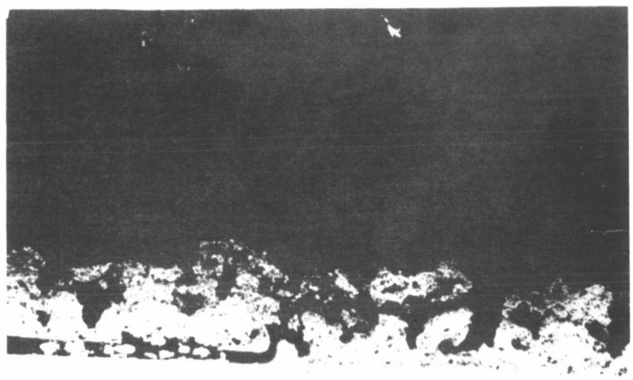


Figure 12

Typical, as polished condition, of specimens from AISI-410 stainless steel sample, coated in accordance with the procedures described in Article 6 of CoDeposition Procedures, page 14.

Plating bath had 150 grams (or more) Mo S_2 powder per liter
Current density = 7000 milliamperes per square inch. This is equal to 1008 amperes per square foot.
Plating time = 45 minutes.

Note that agglomerated Mo S_2 particles form small platelets or stringers, appearing as minute gray sticklike configurations, in the nickel matrix.

670-18 (2/64)

W. G. Grenier (Signature) 4-9-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	ATD	673Y03-14	1200-49

CONCLUSIONS:

The purpose of this study was to determine the feasibility of co-depositing a film of Mo S_2 and Nickel onto a substrate thereby creating a reservoir of Mo S_2 which serves as the lubricant for space components.

Feasibility study type apparatus was set-up in the laboratory and trial-runs were made. The results are shown in the text and they show that the process is feasible and that a number of problems are evident, i.e. (1) optimum particle size, (2) correct current density, (3) particle dispersion in the matrix and uniformity of the co-deposited film.

An investigation of the literature shows that a very limited number of industrial concerns have co-deposited insolubles with a nickel or copper plate and that a number of commercial products–abrasion resistance surfaces for instance–are being produced.

From this evidence, it is shown that the process is feasible but more work has to be done on it to prove its use as a "space" lubricating technique. A following memo will go into this in detail.

C. E. Vest (Signature) 6-12-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	BUILDING	ROOM	PROJECT	JOB ORDER NUMBER	REQUEST NO.
C. E. Vest	11	S-120	MoS_2 In Situ	673Y03-14	1200-41

DATE IN	DATE COMPLETED	PERFORMED BY
8-19-63	2-12-64	W. G. Grenier, J. L. Wall, & J. A. Munford

NAME OF TEST

Mo S_2 In Situ, Adaptation Program, Completion Phase.

DESCRIPTION OF SERVICE OR ARTICLE TESTED:

Service No. 1; Place Mo S_2 in Situ on various component configurations,
Service No. 2; Establish procedure for electrodeposition of the Mo O_3 complex,
Service No. 3; Endeavor to establish plating parameters for reproducible plating thickness of 150×10^{-6} inches $\pm 25 \times 10^{-6}$ inches. Coincident with this evaluate and determine the best possible method for measurement of plating thickness.

EQUIPMENT INVOLVED:

See prior report on S.R. 1200-41 dated 9-13-63 for complete list and photos of cleaning equipment, conversion equipment, and metallurgical Laboratory equipment involved. Goddard publication X-673-64-13, pg. 178.

Special laboratory plating equipment, developed specifically for this phase, is shown in photographs Figure 1, 2, 7 & 8 of the data. The Permascope, Type ESeJ4a Electronic thickness tester, employed as part of service No. 3, is described on sheet no. 44.

RESULTS:

Service No. 1, See Tables Nos. 1 & 2, Sheets 33 & 34, and Tables Nos. 3 & 4, Sheets 40 & 41

Service No. 2, See Sheets Nos. 14-16 incl.

Service No. 3, See Sheets Nos. 20, 25 & 26, see data sheets 42-56 incl.

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS In Situ	673Y03-14	1200-41

PROCEDURE: SERVICES PERFORMED

Three interrelated services were performed under this Service Request. They are as follows:

Service No. 1 – See sheets nos. 3-13 incl. MoS_2 was placed in Situ on several components of different configurations.

Service No. 2 – See sheets nos. 14-16 incl. A positive procedure was established for plating the $MoO_3 \cdot 1/3\ NH_3 \cdot 6H_2O$ molecule on various metals.

Service No. 3 – See sheets nos. 17-26. The feasibility of plating the MoO_3 complex on a material to a thickness of 150×10^{-6} inches plus or minus 25×10^{-6} inches was studied. Also, the best method of plate thickness measurement, optical or electronic, was evaluated.

W. G. Grenier 2-14-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, GENERAL

It was requested that MoS_2 in Situ, be placed on several components of differing configurations. The components consisted of the following: Three, Rex AAA, steel cubes; three, Rex AAA, steel discs; six, SAE-52100 steel, bearing rings; and four, stainless steel, New Departure, ball bearing assemblies, less the bearing balls.

The general procedure for this process is as follows:

1. With plating apparatus, such as that shown in Figures 1 or 2, Sheet No. 27, apply an electrodeposited film of $MoO_3 \cdot 1/3\ NH_3 \cdot 6H_2O$ on an object.

2. Place the plated object, or objects, in a chamber filled with H_2S gas and hold for a period of time under heat and pressure. Apparatus used for this purpose is shown in Figure 3, Sheet No. 28.

In electroplating, the current requirements are generally expressed in units of electric current flow through the plating cell, per unit of surface area being plated. Then, total current requirements, for any one plating cycle, is dependent on the total surface area to be plated at that time. Therefore each component's dimensions were carefully determined by means of micrometer calipers and the surface areas calculated. Configurations for all components are given in the data in Figures 4-12, Sheets 29-38.

For purposes of this report, the word conversion, is defined as that process whereby the MoO_3 complex plating on an object is converted to MoS_2 in Situ on that object. Conversion records of the plated, Rex AAA steel cubes and discs, and the SAE-52100 steel rings, are presented in Tables Nos. 1 & 2, Sheets Nos. 33-34, in the data. Included as a footnote to the

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, GENERAL (Continued)

tables, are the past conversion appearance of the objects. Conversion records, for the plated stainless steel bearing components are given in Table No. 3, Sheet No. 40, of the data. Post conversion appearance of each component is given separately in Table No. 4, Sheet No. 41, of the data.

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, COMPONENT, PREPLATE CLEANING

Two different types of detergents were used as cleaning agents for objects about to be plated. Triton X-100 was used in water to partially clean the cubes, discs, and rings of Figures 4-6 inclusive. Fisher Sparkleen, detergent in water, was used to partially clean the stainless steel New Departure Bearing assemblies of Figures 9-12 inclusive.

Ultrasonic cleaning apparatus was used exclusively in cleaning each object to be plated. Each component, or group of components, was placed in a small beaker containing the appropriate cleaning agent. The small beaker was then placed in the larger ultrasonic transducer beaker, which had previously been partially filled with water, for a given time period. The particular preplate cleaning procedure used for this series of components was as follows:

1. Clean in detergent solution – 3 minutes
2. Rinse thoroughly in hot tap water
3. Rinse in Methanol or Propanol
4. Clean in Trichloroethane – 3 minutes
5. Clean in Benzene – 1 to 3 minutes
6. Remove from Ultrasonic cleaner, but retain components in Benzene until ready to activate the surface for plating.
7. Remove from Benzene with forceps or tongs.
8. Place component, or components, in plating clamps
9. Dry in air blast

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, COMPONENT, PREPLATE CLEANING (Continued)

10. Immerse immediately in Activation solution.

670–15

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, ACTIVATION

I. Procedure for Components in this series:

1. Prepare 20 v/o H_2SO_4 in distilled water.
2. Heat solution to 180°F.
3. Immerse in hot solution for 15-30 seconds.
4. Rinse in distilled water.
5. Place immediately in plating bath and start plating.

Note: It is not abnormal for objects to appear smutty black after activation.

II. Precautions in preparation of H_2SO_4 Activation bath:

When mixing concentrated H_2SO_4 with water, a large quantity of heat is generated by the reaction. Therefore, it is recommended that the H_2SO_4 be poured into a relatively small quantity of water first. The dilute solution should then be cooled sufficiently so the container may be handled comfortably by hand. The dilute acid may then be poured into the larger quantity of water with little danger to the careful technician.

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, PLATING OF MoO_3 COMPLEX

I. Plating Bath Formula:

1. Distilled water 2000 ml
2. Ammonium Formate (NH_4COOH) 109.2 gms
3. Molybolic Acid (85%) 25.2 gms

II. Preparation of Plating Bath:

1. Place 2000 ml distilled water in a battery jar
2. Add 109.2 gms ammonium formate (NH_4COOH)
3. Place on Tempco Stirrer Hot Plate and agitate
4. Set temperature control on medium
5. Add 25.2 gms Molybdic Acid (85%)
6. Stir and heat to approximately 82°C, putting item 5 in solution.
7. Let cool to room temperature before using

III. Notes on Plating Bath Color Changes:

During the heating phase of preparation, it was observed that the plating solution shifted from clear to a brackish green color at 80°C. Upon cooling, with the Molybolic Acid in solution, the bath frequently turns a deep teal blue. During use, the bath may either deepen in color or tend to become clearer. The Originator contacted Dr. Trzeciak of I.B.M. with regard to this phenomenon. Dr. Trzeciak stated, that the final color of the plating solution depends on the valence of the Mo ions at the moment. He further stated, that a room

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, PLATING OF MoO_3 COMPLEX (Continued)

temperature color of either blue, or clear, is indicative of a good bath for plating of the $MoO_3 \cdot 1/3\ NH_3 \cdot 6H_2O$ moledule.

IV. Holding of Objects for Plating:

It was considered desirable that all objects plated be secured in the plating bath, such that each of a given shape be orientated similarly. A unique, specimen holding, electrode was developed and used for this purpose. It is shown in Figures 1, 7 & 8 of the data. By means of this type of electrode, several objects can be plated simultaneously with identical orientation. Objects may also be plated individually with any desired orientation reproducible from one plating series to another. The holding clips can be changed to suit the individual needs of the plater. In plating the large discs and cubes, the small clips shown in Figures 7 & 8 were replaced with large battery clips, used normally in automobiles.

V. Power Supply:

1. In past experimentation on this project, the Buehler Micromat Etcher was used as a D.C. power source and current control. It was considered desirable to have a more precise current control system. Therefore the nine volt battery and milliammeter system shown in Figure 1, was devised. By switching to a meter with the ideal scale and appropriate variable resistance, it was possible to control the current through the plating cell very accurately. This system was used exclusively in plating the MoO_3 complex on the components shown in Figures 4-8, inclusive. The components appeared to plate with a smooth, black plating, at the current density of 12 ma per square inch, for a plating time of 6 minutes. The MoO_3

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, PLATING OF MoO COMPLEX (Continued)

complex plate also appeared to convert to the MoS in Situ properly. However other items did not appear to coat properly.

2. Some stainless steel bearing races, not mentioned elsewhere in this report, were plated and put through the conversion process with very poor results. The opening series of specimens used in the tolerance feasibility (Service No. 3) portion of this report, showed plating thicknesses considerably less than anticipated. The indication was that a heavier plate was produced using the Micromet Etcher Power supply, than was produced using the battery supply, with the same apparent current density. Therefore, experimentation was conducted with the Micromet Etcher power supply. It was discovered that the milliammeter in this unit was in error by a precise factor of ten (10). This showed that past experiments, believed to have been conducted at 12 ma per square inch, were in reality conducted at 120 ma per square inch. All interested parties were immediately notified and corrections made in reports previously written.

3. During the experimentation with the Micromet Etcher power supply, the battery-milliammeter system was cannibalized. Therefore the Buehler Electropolisher power supply was used throughout the balance of tests conducted in this project. The appropriate milli-ammeter was placed in the plating circuit for each plating cycle. This arrangement is shown in Figure No. 2, Sheet No. 27, of the data.

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, PLATING OF MoO_3 COMPLEX (Continued)

VI. Plating of Objects With MoO_3 Complex:

1. Prepare plating bath as in Article II
2. Use Platinum annode
   2.1 Throughout this project a Pt plated Ti mesh was used to line the vertical sides of a battery jar plating cell.
3. Suspend components in plating bath, using appropriate holder
4. Connect component holding electrode to the negative side of the power supply.
   4.1 Milliammeter should be in this circuit.
5. For the cubes, discs and rings of Figure 4-8 inclusive; Plate at a current density of 12 ma per square inch for 6 minutes.
6. For the New Departure bearing assembly components of Figures 9-12 inclusive; Plate at a current density of 24 ma per square inch, for 12 minutes.
7. Rinse in distilled water.
8. Air dry in room temperature air blast.
9. Wrap, like a sandwich, in Aluminum foil.
10. Place in Conversion Chamber within 90 mimutes of removal from plating bath. This is an empirical time limit, arrived at by personnel of I.B.M.

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, CONVERSION

The conversion apparatus used throughout this project is shown in Figure 3. The conversion procedures followed for all components, is as follows:

1. Within 90 minutes after plating, place wrapped object in the conversion chamber.
2. Place 2S Aluminum 'O' ring in its groove.
3. Secure the face plate, containing the manifold.
4. Attach KOH filter tube to the inner hose bib.
   4.1 Be certain that the valve is closed securely.
5. Open valve at rear of pressure chamber
6. Attach vacuum pump hose to forward hose bib.
   6.1 Open the valve.
7. Evacuate chamber and close the forward valve.
8. Open all valves on the H_2S gas cylinder and fill chamber.
9. Check for leaks
10. When the pressure inside the chamber has stabilized at 250 + psi, shut all valves in back of the chamber, including those on the H_2S gas cylinder.
11. Turn on Variacs in heating tape circuits.
12. Adjust the Variacs such that each heating tape circuit is drawing the same current.
    12.1 Current should be approximately 2 amperes
13. When the chamber pressure has built up to 390 psi, reduce the current in the heating tape circuits. This reduction will be judged by the rate of chamber pressure increase,

W. G. Grenier 12-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 1, CONVERSION (Continued)

from the original 250 + psi to the operating level of 390 psi. Generally the reduction will be approximately 0.7 amperes.

14. Hold pressure for the desired time, by adjusting the current flow through the heating tapes.

    14.1 Past experimentation indicates that four hours is adequate.

15. After prescribed time at temperature and pressure, open the inner hose bib valve, on the front manifold and slowly bleed gas from the chamber through the KOH filter system.
16. Open valve at rear of conversion chamber.
17. Connect a tank of innert gas to the forward hose bib, by means of the flexible tuging.
18. Flush system several times with innert gas, filtering residual H_2S gas through the KOH filter.
19. Disconnect gas
20. Remove the face plate, with manifold, from the conversion chamber.
21. Remove and observe coated objects.

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 2

It was desired to establish a positive procedure of electroplating the $MoO_3 \cdot 1/3 \cdot 6H_2O$ molecule on various metals. For this purpose the experiences encountered in performance of Service No. 1, this report, and a review of the results of all past work on this project indicated the following procedures to be correct.

I. Cleaning – Any Metal:

A. Equipment required,

1. Ultrasonic cleaning apparatus
2. Clock or interval timer
3. Beakers, or similar containers for solutions
4. Sink with hot and cold water

B. Procedure for Ultrasonic cleaning

1. Immerse in detergent and water – 3 minutes
2. Rinse, hot water followed by Propanol
3. Immerse in Trichloroethane – 3 minutes
4. Immerse in Benzene – 1 minute
5. Remove from Ultrasonic cleaner, but retain objects in Benzene until prepared to activate

II. Activation, various metals;

A. Mild, plain carbon, steels

1. Immerse in 50% HCL

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 in Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 2 (Continued)

2. Hold at room temperature - 30 seconds to 1 minute

3. Rinse briefly in free flowing tap water

4. Plate immediately

B. All Aluminums

1. Immerse in 20% NaOH, at 180°F for 30 seconds

2. Rinse and plate as for mild steel

C. All stainless steels

1. Immerse in 20% H_2SO_4 in distilled water

1.1 Follow precautions outlined on Sheet No. 7 of this report.

2. Hold in solution at 180°F for 15 seconds

3. Rinse and plate as for mild steel

III. Plating, all materials;

A. Equipment required,

1. D. C. power supply with precise controls

2. Milliammeter in appropriate range

3. Various chemicals for plating bath

4. Container for plating bath

4.1 In this project a 2400 ml capacity battery jar was used

5. Platinum electrode, must circumbscribe object(s)

5.1 Connect to the negative side of power supply

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 2 (Continued)

6. Clamp type electrode to hold object(s) in plating bath

   6.1 Connect to the negative side of power supply

   6.2 Coat clamps with passive insulating material

7. Agitation device for plating bath

   7.1 Recommend magnetic type stirrer for laboratory use

8. Distilled water, in containers for rinsing

9. Air blower, for drying plated object(s).

B. Plating bath formula:

1. 2000 ml, distilled water

2. 109.2 gms, NH_4COOH

3. 25.2 gms, 85% Molybolic Acid

4. Prepare as per paragraph II, Sheet 8 of this report.

C. Current and Time Requirements

1. Maintain a current density of 12 ma per square inch

2. Plating time will vary, depending on the plate thickness desired. For probable plate thickness, resulting from a given plating time see Figures 17-19 inclusive, Sheets Nos. 54-56 inclusive.

W. G. Grenier 2-14-63
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, GENERAL, MoO_3 PLATE THICKNESS STUDY

I. Objectives

It was desired to determine the feasibility of plating a predictable thickness, of 150×10^{-6} inches, of the MoO_3 complex on a material. It was believed that this could be accomplished by control of plating time. Therefore, it was further desired that the plate thickness be predictable as a function of plating time, to within twenty-five millionths of an inch. The desired accuracy, presupposes a uniform current density of 12 ma per square inch of surface being plated. Coinciding with the thickness application problem, it was desired to determine the best method of plate thickness measurement, i.e. optical or electronic.

II. Specimen Selection

Since the experiments were to be considered developmental, all specimens used were as near identical as is practical. The specimens, fabricated from AISI-410 stainless steel, are described in detail on Sheet No. 42, of the data. Typical specimens, both before and after plating are shown in Figures 13 and 14 of the same Sheet. A specimen numbering system, starting with the number 100, was chosen, to avoid confusion with earlier work on this project. Specimens, numbered 100 through 126, were plated in groups of three, using the apparatus shown in Figure 1, Sheet No. 27. All other specimens were plated, using the apparatus shown in Figure 2. Plating procedures were as described under Service No. 2, Sheets numbered 14-16 inclusive.

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, PLATE THICKNESS STUDY (Continued)

III. Plating Thickness Measurements

After plating, specimens numbered 100-126 inclusive were prepared for optical measurement of the plating thickness. Those specimens were mounted on edge in glass filled epoxy and polished for metallographic observation. Detailed mounting and polishing procedures are dealt with, later in this report. Each specimen was observed at magnifications up to 2200 diameters, using the Bausch & Lomb Research Metallograph. No plating was discernable, as is shown in Table 5, Sheet 43, of the data. Additional specimens, numbered 127-140 inclusive, were then plated in groups of two. Each group was plated for a different length of time. The even numbered specimens, from this series, were submitted to Mr. J. A. Munford of the Fabrication Division, for plating thickness measurements using the Permascope. The odd numbered specimens were retained for optical determination of the plate thicknesses. Results of both methods are given in Tables 6 and 7, Sheets 45 and 46, of the data. That data indicates, the Permascope to be capable of measuring a thinner coating than can be defined by even the best optical means. The data also indicated that to achieve the desired plate thickness, the specimens should be plated for a period of time approximating 24 minutes. Further, for those plating times of less than 24 minutes, the Permascope indicates a thinner plate than does the optical data. This is not entirely unexpected and problems involved in optical measurements are dealt with later in this report; see Sheets Nos. 25 and 26.

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-4	1200-41

PROCEDURE: SERVICE NO. 3, PLATE THICKNESS STUDY (Continued)

IV. Permascope Description

The Permascope is an electronic thickness measuring device for thin films. It measures the average coating thickness over a ten millimeter distance. It measures the thickness of nonmagnetic materials, plated over magnetic substrates, or vice versa. It is a nondestructive measuring instrument and is described in detail on Sheet 44 of the data.

V. Evaluation of Plate Thickness Reproducability

To evaluate the reproducability of a given plate thickness, with respect to a given plating time, twenty-four (24) additional specimens were plated. They are numbered 141-164, inclusive. A flow chart, showing the disposition of each specimen is given as Table No. 8, Sheet 47, of the data. Note that two (2) plating baths are mentioned, in Old Bath and New Bath. The term, Old Bath, refers to that plating bath used throughout the earlier portions of this test series. The term, New Bath, refers to a plating bath prepared expressly for this phase of the tests. The original chemical compositions of both plating baths were identical and were in accordance with Service No. 2, Item III-B, Sheet 16.

After plating, in accordance with Table 8, all specimens were submitted to Mr. J. A. Munford for Permascope measurements of the plating thickness. The Permascope data is reported in Table 9, Sheets 48 and 49, of the data. All specimens were returned, after the Permascope measurements, with no apparent damage to the coating. Therefore, five (5)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, PLATE THICKNESS STUDY (Continued)

specimens, selected at random, were mounted in green bakelite for comparative optical measurement of the plating thickness. Green bakelite was chosen because, it is harder than black bakelite and simpler to use than glass filled epoxy. The thickness of plating, as determined by this method is presented in Table 10, Sheet 50 of the data. Photomicrographs of two typical specimens are presented at a magnification of 250 diameters, in Figures 15 and 16, Sheet 51, of the data. Note that in spite of optimum care during the mounting and polishing of the specimens, the mounting medium still proved inadequate. Other specimens were therefore mounted in glass filled epoxy and polished for optical measurement of the plating thickness. Due to a failure of the epoxy to cure properly, accurate optical measurements of those specimens was not possible. A brief synopsis of both Permascope and optical measurement data, is presented in Table 11, Sheets 52 and 53 of the data.

The Permascope data is presented in graphic form in Figures 17 through 19, Sheets 54-56 inclusive of the data. While the desired range of plating thickness was not actually achieved, Figure 19, Sheet 56, indicates the feasibility of probable achievement.

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, OPTICAL MEASUREMENTS OF PLATING THICKNESS

I. Specimen Mounting, Standard Procedures

A. Mounting with powdered bakelite, black or green

1. Use the Buehler Powermet Mounting Press
2. Locate specimen on piston, such that desired face is in contact with piston face
3. Lower piston to bottom of the cylinder
4. Pour 30 ml powdered bakelite into cylinder, over and around the specimen
5. Secure the cylinder top clamp
6. Push 'up' button on pump control
7. Adjust piston pressure to read 3300-3500 psi
8. Place heaters around cylinder
9. Maintain temperature of 150°C for minimum of 5 minutes
10. Remove heaters and replace with cooling blocks
11. When temperature has dropped to at least 100°C release piston pressure and remove specimen
12. Label the mount immediately with the appropriate specimen designation.

B. Mounting in glass filled epoxy

1. Spray a sheet of aluminum, or thin steel, with Fluorocarbon, or other similar dry lubricant. Place a specimen, with the desired face down, in the center of a previously marked bakelite ring form. Pour the prepared glass filled epoxy over and around the

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, OPTICAL MEASUREMENTS (Continued)

specimen, until the ring form is filled. Set to one side and allow mount to cure for at least 24 hours.

2. To prepare the glass filled epoxy mounting medium: Mix, by weight, three (3) parts Hysol 2038 epoxy with two (2) parts, 325 mesh, ground glass. Allow the mixture to stand, at least 24 hours, to eliminate trapped air bubbles. Combine the glass and epoxy mix with Hysol 3404 Hardener in the weight ratio of fourteen (14) parts epoxy and glass mix to one (1) part hardener. Stir until thoroughly blended. Failure to mix thoroughly will result in soft spots in the interior of the mount.

3. In those specimens selected from Table 8, to be mounted in this medium, difficulty was encountered, in that the epoxy did not cure properly. The problem seems to be chronic with this particular epoxy, however no evaluation has been attempted. The problem, not immediately apparent, became obvious in the polishing operations. Therefore, all specimens were placed in the laboratory heat treating furnace and held at 100°C for 24 hours. Following this, baking out process, the mounts appeared properly cured, so the polishing procedures were followed accordingly. It now became evident that, due to shrinkage of the epoxy, the plated MoO_3 complex was no longer protected by the mounting medium. Therefore no reliable optical measurements were possible. It should be noted, that much

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, OPTICAL MEASUREMENTS (Continued)

past work, utilizing the glass filled epoxy for edge retention, has shown it to be a superior medium, when proper curing is effected.

II. Polishing of All Specimens

Bakelite mounted and glass filled epoxy mounted specimens were polished in identical manners. Following established procedures, for edge study, each specimen was first rough-ground on the wet belt surfacer, using 80 and 120 grit silicon carbide belts. Each specimen was ground sufficiently, to be in an area well away from corner plating effects. Polishing was accomplished on the three (3) wheel, slow speed, lapping table, equipped with Automet attachments for each wheel. Copious quantities of water were used on the papers. A load setting of from 40 to 50 pounds was used on the Automet, with the maximum time of five (5) minutes running time for any step. Either Buehler Texmet, or silk cloth, was used on the diamond charged wheels, for the intermediate polishing steps. Final polish was accomplished using Gamal on a well dressed microcloth lap.

III. Measuring the Plated MoO_3 Complex

Following the final polish with Gamal, each specimen was observed under the microscope. For purposes of optical measurements, the Bausch & Lomb Research Metallograph was used. A 50 X, appochromatic objective, with an N.A. of 95.0, was used in conjunction with a Filar micrometer eyepiece. In this combination, each filar unit is equal to 2.63×10^{-6}

W. G. Grenier 2-14-64
(Signature) *(Date)*

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, OPTICAL MEASUREMENTS (Continued)

inches. It was impossible to obtain data from those specimens mounted in the epoxy. The data from those specimens mounted in the green bakelite would be seriously questioned, were it not for the Permascope evidence, see Table 11, Sheets 52 and 53.

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, DISCUSSION OF MEASUREMENT METHODS

I. Problems in Optical Measurement Methods

1. The method is complicated and fundamentally destructive.
2. Accuracy, demands the subject be sectioned and mounted, such that the observed plane is one which is normal to the plane tangent to the coated surface, at the precise point of observation.
3. Existing specimen mounting mediums and polishing techniques, leave much to be desired in both, true edge retention of soft porous coatings and flatness in polished specimens.
4. In soft, porous coatings, drag-out may occur during the polishing operations. Therefore it is frequently necessary to measure the width of the void, where the coating had been, to help determine the original plate thickness.
5. Slight drag-out, may cause sub-microscopic rounding of edges, producing a very minute halation effect in the microscope. This may be unobserved, thereby introducing error.
6. The Filar hair, used in the filar micrometer eyepiece, is itself two plus (2+) filar units thick. Therefore extreme care and patience is required to obtain accurate data.
7. The foregoing points up one common factor, i.e. Technician proficiency and dedication is mandatory.

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

PROCEDURE: SERVICE NO. 3, MEASUREMENT METHODS (Continued)

8. Summation

In measuring a plating thickness by optical methods the areas for human error are many. The technical integrity of the Technician is of paramount importance.

II. Permascope Measurement Method, Advantage of

1. The procedure is nondestructive.
2. No special mounting or polishing is required
3. Unique specimen alignment is not required
4. The element of human error is reduced to a nearly negligible factor. It must be remembered that the instrument should be calibrated frequently to assure its accuracy.

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: ELECTRODEPOSITION APPARATUS

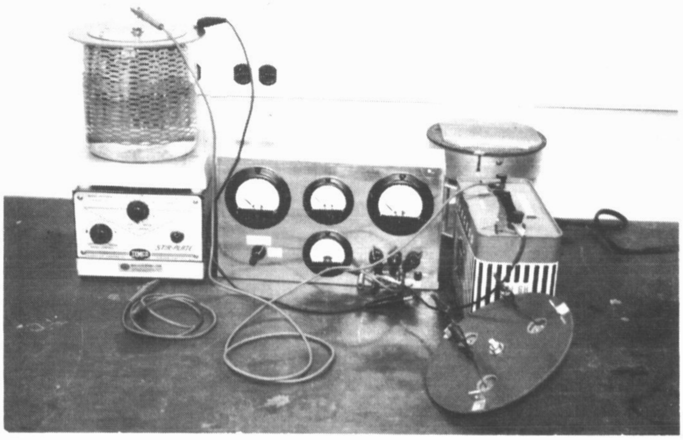


Figure 1

Photograph of plating apparatus employed in the first stages of this test series. Included are the unique sample holders and precise control power supply.

Figure 2

Photograph of laboratory plating apparatus employed in Service items 3 & 4, Sheet 1. Note that the dry battery shown in Figure 1 has been replaced by the Electropolisher power supply.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: CONVERSION APPARATUS

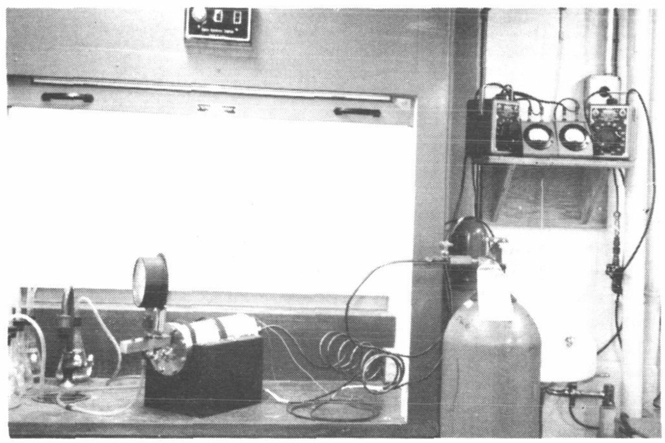


Figure 3

Pictured here is the conversion chamber and support equipment used throughout this program. The apparatus is used to convert electroplated MoO_3 complex to MoS_2 in Situ.

Figure 3, from left to right: KOH absorption system, for bleeding of H_2S gas from the conversion chamber; Conversion chamber, with pressure gauge mounted on the front, and wound with two lengths of heating tape; and copper tubing, leading to the high pressure cylinder of H_2S gas. In the upper right hand corner can be seen the two Variacs and associated ammeters, controlling power input to the heating tapes.

A standard laboratory vacuum pump (not shown) is used to evacuate air from the conversion chamber prior to filling with H_2S gas.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: COMPONENT CONFIGURATION; SERVICE ITEM NO. 1

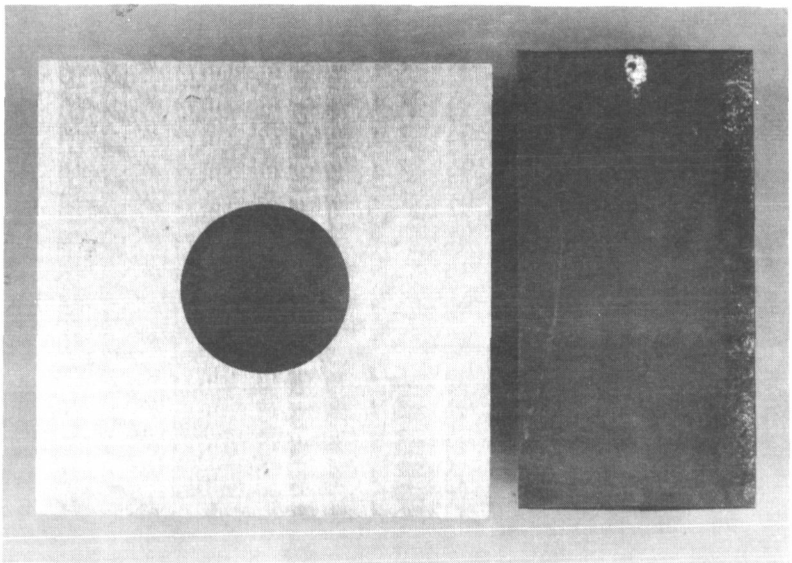


Figure 4

Photograph of Rex AAA tool steel cube, as received for placing of MoS_2 in Situ.

Surface Area Calculations

$$A \text{ total} = 2a_1 + 4a_2 + a_3 = 6.184 \text{ square inches}$$

Where:

a_1 = Exposed face area
a_2 = Exposed side area
a_3 = Surface of cylindrical center hole.

Total plating current at 12 ma/ins^2 = 74.2 ma.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: COMPONENT CONFIGURATION; SERVICE ITEM NO. 1

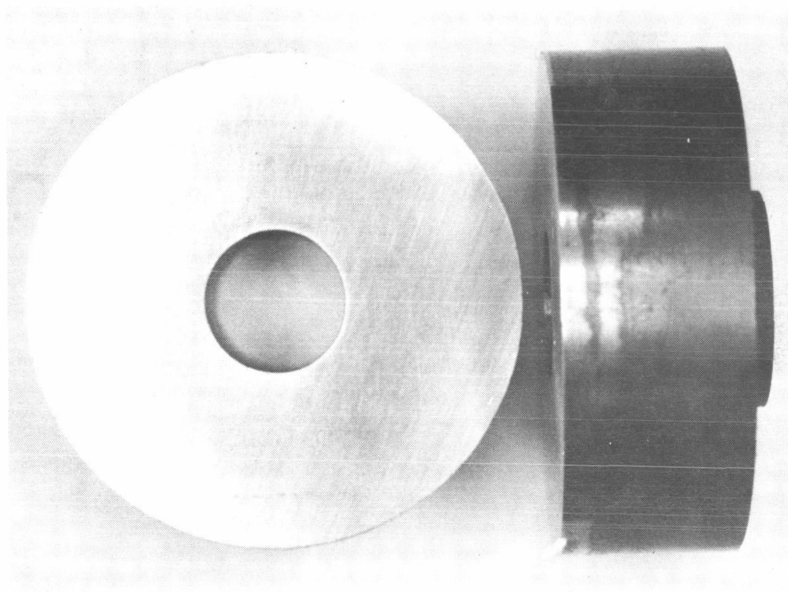


Figure 5

Photograph of Rex AAA tool steel disc, as received for placing of MoS in Situ.

Surface Area Calculation

$$A\ \text{total} = a_1 + 2a_2 + a_3 + a_4 = 8.830 \text{ square inches}$$

Where:

a_1 = Exterior cylindrical surface area of hub
a_2 = Exposed face area
a_3 = Large exterior cylindrical surface area
a_4 = Center hole interior cylindrical surface area.

Total plating current at 12 ma/ins^2 = 105.96 ma.

670-18 (2/64)

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: COMPONENT CONFIGURATION; SERVICE ITEM NO. 1

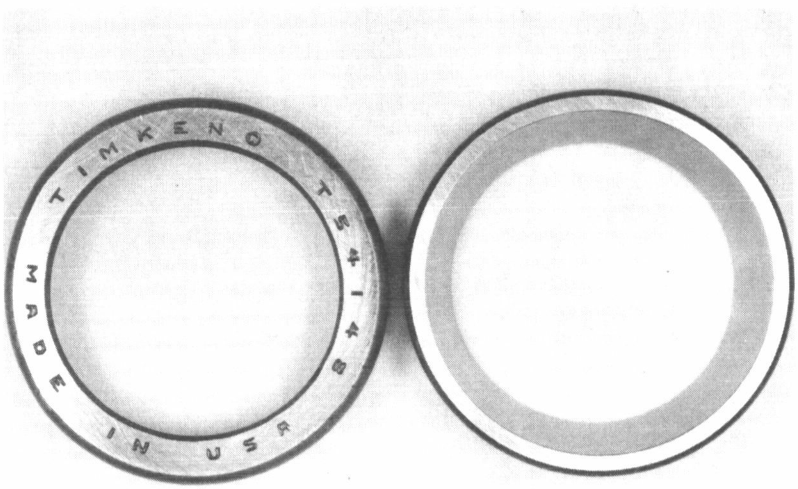


Figure 6

Photograph of SAE-52100 steel rings, as received for placing of MoS_2 in Situ.

Surface Area Calculation

$$A \text{ total} = a_1 + a_2 + a_3 + a_4 = 3.693 \text{ square inches}$$

Where:

a_1 = Backside face, surface area
a_2 = Front face, surface area
a_3 = Interior conical surface area
a_4 = Exterior cylindrical surface area

Total plating current at 12 ma/ins^2 = 44.3 ma per ring.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: S.A.E. 52100 STEEL RINGS, PREPARATORY TO PLATING, SERVICE ITEM NO. 1

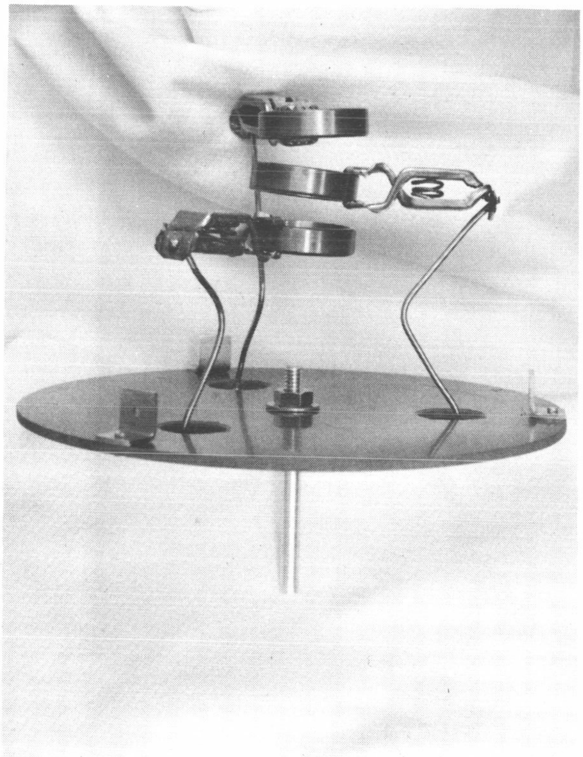


Figure 7

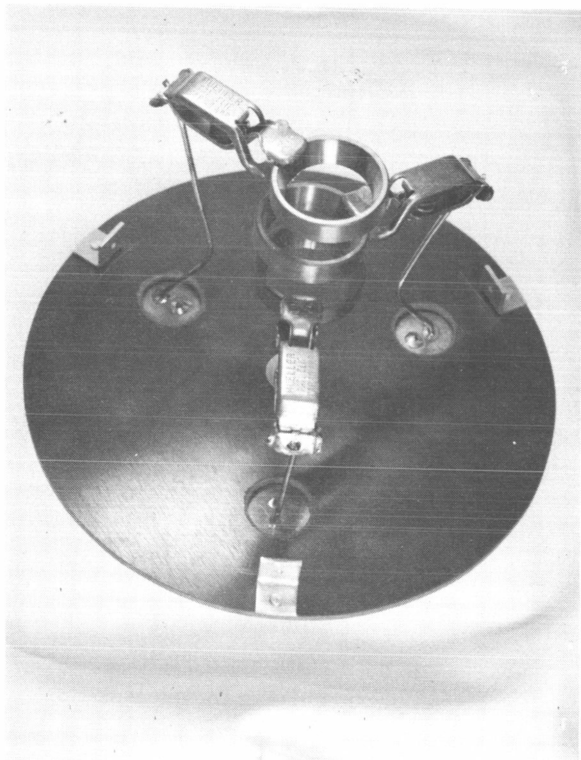


Figure 8

Photographs showing the SAE 52100 steel rings in laboratory sample holder, preparatory to plating with MoO_3 complex. Sample holder is same as that shown in Figure 1.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA:

Table 1

CONVERSION CONDITIONS, 3 DISCS – FIG. 5, & 1 CUBE – FIG. 4

	TIME O'CLOCK	CHAMBER PRESSURE P.S.I.	HEATING TAPE CONDITIONS			
			AMPERES		VOLTAGE	
			#1	#2	#1	#2
	1008	H_2S Gas turned on				
	1010	252	2.2	2.2	95	70
	1016	280	2.2	2.2	95	70
	1021	300	2.2	2.2	95	70
	1026	325	2.2	2.2	95	70
	1030	350	2.2	2.2	95	70
CONVERSION START	1036	375	2.2	2.2	95	70
	1040	390	1.5	1.5	65	45
	1100	400	1.5	1.5	65	45
	1110	402	1.3	1.3	58	41
	1130	400	1.3	1.3	58	41
	1200	395	1.4	1.4	60	43
	1230	396	1.4	1.4	60	43
	1300	397	1.4	1.4	60	43
	1330	395	1.4	1.4	60	43
	1400	390	1.4	1.4	60	43
	1430	390	1.4	1.4	60	43
	1440	390 End of conversion. Flushed system with Nitrogen.				

Upon removal from the conversion chamber the components appeared to have a black adherent coating.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA:

Table 2
CONVERSION CONDITIONS, 2 CUBES – FIG. 4, & 6 RINGS – FIGS. 6-8

TIME O'CLOCK	CHAMBER PRESSURE P.S.I.	HEATING TAPE CONDITIONS			
		AMPERES		VOLTAGE	
		#1	#2	#1	#2
0934	H S Gas turned on				
0936	260	2.1	2.1	89	63
0940	300	2.1	2.1	89	63
1005	360	2.1	2.1	89	63
1006	365	2.2	2.2	93	66
1010	390	1.4	1.4	60	43
1030	399	1.4	1.4	60	43
1100	400	1.4	1.4	60	43
1130	397	1.4	1.4	60	43
1200	390	2.0	2.0	85	60
1205	400	1.4	1.4	60	43
1230	396	1.4	1.4	60	43
1300	390	1.6	1.6	68	49
1332	390	1.6	1.6	68	49
1400	390	1.6	1.6	68	49
1410	390	1.6	1.6	68	49
1410	390 End of conversion. Flushed system with Nitrogen.				

Upon removal from the conversion chamber the components appeared to have a black adherent coating.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: STAINLESS STEEL BEARING ASSEMBLY, DESCRIPTION

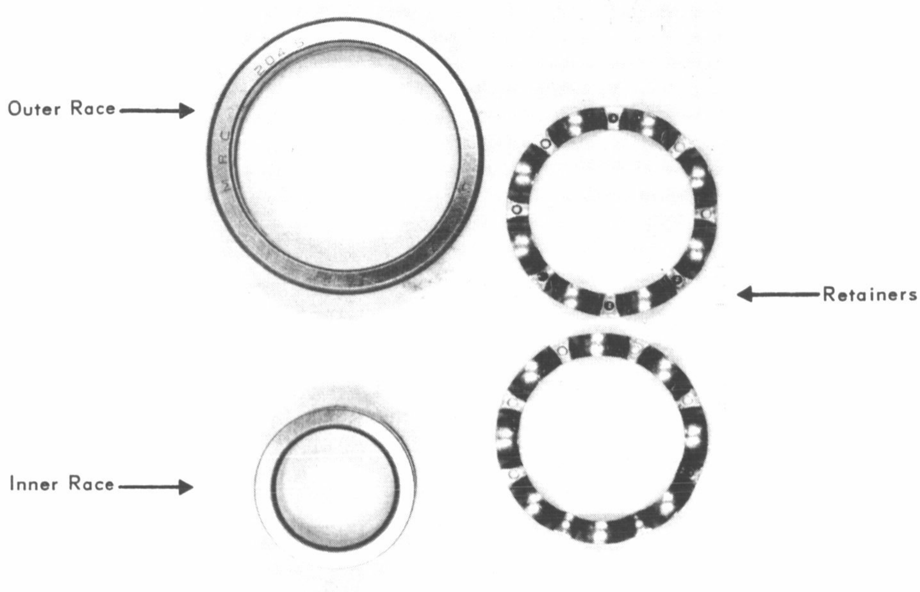


Figure 9

Typical of 4 bearing assemblies, as received, to be coated with MoS_2 in Situ.

PARAMETERS

MoO_3 complex, plating current density	24 ma/ins^2
MoO_3 complex, plating time	12 min.
Conversion time	4 hours.

Components to be in conversion chamber within ninety (90) minutes of removal from plating bath.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: BEARING ASSEMBLY, CURRENT REQUIREMENT CALCULATIONS

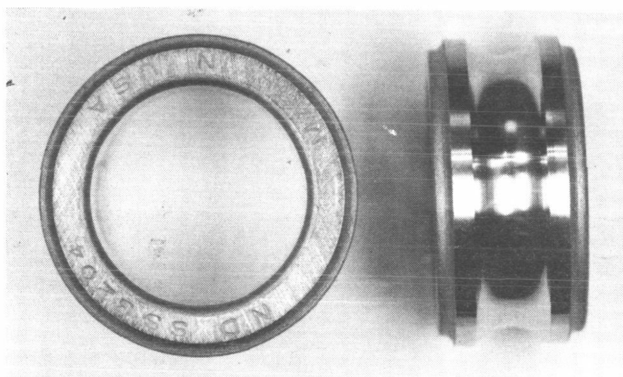


Figure 10–Inner Race Configuration. 1.6X

Exterior treated as a smooth surface.

Min. I.D. = 0".792 Thickness = 0".548
Max. O.D. = 1".070

Areas and circumferences of circles from tables in The Handbook of Physics and Chem.

Where Dia. = 0".792 Area = 0.493 ins^2, Circ. = 2.49 ins.
Where Dia. = 1".070 Area = 0.899 ins^2, Circ. = 3.36 ins.

then $a_1 = 0.899 - 0.493 = 0.406$ ins.^{2} = area of one face $2a_1 = 0.812$ ins.
$a_2 = (0.55)(3.36) = 1.85$ ins.^{2} = area outer surface
$a_3 = (0.55)(2.49) = 1.37$ ins.^{2} = area inner surface.

Total surface area: $A = 2a_1 + a_2 + a_3$
$= 0.82 + 1.85 + 1.37 = 4.04$ ins.^{2}
Current density = 24 ma/ins.^{2}
Total current: $I = (24)(4.04) = 97$ ma.

670-18 (2/64)

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: BEARING ASSEMBLY, CURRENT REQUIREMENT CALCULATIONS

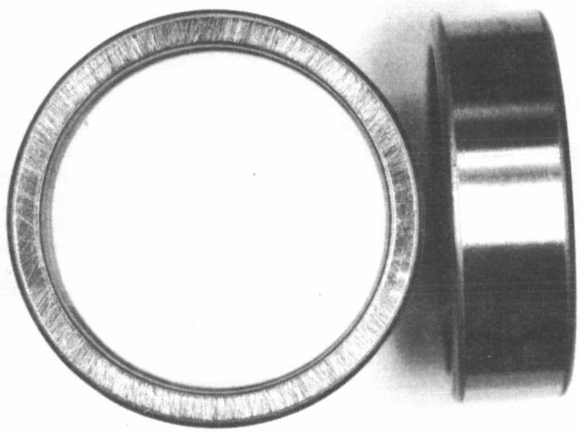


Figure 11—Outer Race Configuration. 1.2X

Interior treated as smooth flat surface

Min. I.D. = 1".516 Thickness = 0".530
O.D. = 1".851

Areas and circumferences of circles from The Handbook of Physics and Chemistry.

Where dia. = 1".516, Area = 1.81 ins.^{2}, Circ. = 4.78 ins.
Where dia. = 1".851, Area = 2.69 ins.^{2}, Circ. = 5.81 ins.

Then: a_1 = 2.69 − 1.81 = 0.78 ins.^{2}, area of one face, $2a_1$ = 1.56 ins.
a_2 = (0.530) (5.81) = 3.07 ins.^{2}, area of outer surface
a_3 = (0.530) (4.78) = 2.53 ins^2, area of innter surface

$A = 2a_1 + a_2 + a_3 = 7.16$ ins.^{2}, total surface area

Current density = 24 ma/ins.

Total current: I = (24) (7.16) = 172 ma.

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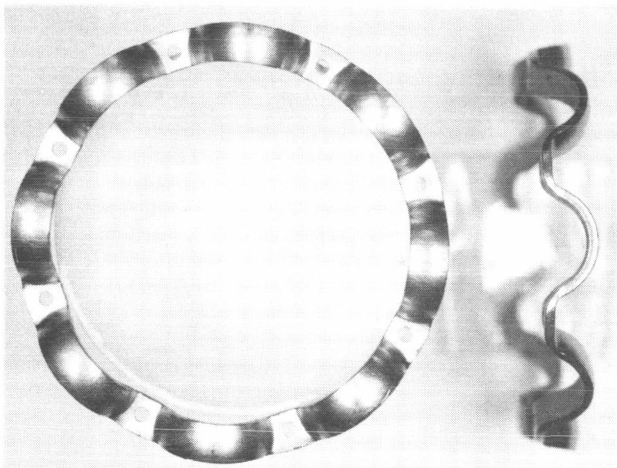
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: BEARING ASSEMBLY, CURRENT REQUIREMENT CALCULATIONS



1.7X
Figure 12–Retainer configuration. Two retainers are used in each assembly.

Since no simple, accurate, method of determination of the surface area for this configuration was readily available, the following method was employed.

A piece of waxed cord was impressed around the shape and held such that it was approximately centered between the I.D. and O.D. The string was taken once around the shape, removed and the indicated length 'L' measured on a standard scale. This value is taken to be the mean circumference of the retainer.

L = 5.00 inches, Stock thickness: T = 0.014, Width: W = 0.132

Total surface area: $A = 2WL + 2TL$
$A = 1.32 + 0.14 = 1.46$ ins.^{2}

Current density $= 24$ ma/ins.^{2}

Total current per retainer: $I = (24)(1.46) = 35.1$ ma.

Total current for pair: $I_p = (2)(35.1) = 70.2$ ma.

670-18 (2/64)

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SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: Bearing Assembly, Current Requirements.

Total current required to plate the MoO_3 complex on one complete assembly.

Outer Race = 172 ma
Inner Race = 97 ma
Retainers (Pair) = 70 ma

Total = 339 ma

Add 5% to total current, to allow for holding clips and bearing raceway areas not included in surface area determinations.

(339 ma) (0.05) = 16.95 ma
339 ma + 16.95 = 355.95 ma or 356 ma

Plate at 356 ma or 0.36 amperes for 12 minutes.

Each assembly was plated at 0.36 amps, D.C.

Post plate condition of each component of each assembly was unusually good. Each part was very black and very glossy in appearance. The plating appeared to be tight with no bubbles or flakes.

1st assembly was out of plating both at 1144 hours.

670-18 (2/64)

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SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA:

Table 3
BEARING ASSEMBLIES, CONVERSION CONDITIONS

First assembly out of plating bath at 1144 hours
All assemblies in pressure chamber and under vacuum at 1310 hours
H_2S gas in pressure chamber at 1323 hours

	TIME O'CLOCK	CHAMBER PRESSURE P.S.I.	HEATING TAPE CONDITIONS			
			AMPERES		VOLTAGE	
			TAPE 1	TAPE 2	TAPE 1	TAPE 2
	1323	H S Gas turned on				
	1325	250	2.5	2.5	105	75
	1330	300	2.5	2.5	105	75
	1343	375	2.5	2.5	105	75
Conversion Start →	1352	390	1.5	1.5	65	45
	1355	395	1.5	1.5	65	45
	1400	396	1.5	1.5	65	45
	1410	402	1.0	1.0	45	30
	1430	395	1.3	1.3	58	40
	1445	390	1.5	1.5	65	45
	1500	400	1.5	1.5	65	45
	1530	400	1.5	1.5	65	45
	1600	394	1.6	1.6	69	48
	1630	394	1.6	1.6	69	48
	1700	395	1.6	1.6	69	48
	1730	395	1.6	1.6	69	48
	1745	395	1.6	1.6	69	48
	1752	395 End of conversion. Turn off current to heat tapes. Bleed gas from chamber and flush system with Helium.				

Remove assemblies the following morning at 1100 hours.

670-18 (2/64)

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SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS In Situ	673Y03-14	1200-41

DATA:

Table 4
POST CONVERSION APPEARANCE OF BEARING ASSEMBLY COMPONENTS

ASSEMBLY NO.	COMPONENT DESCRIPTION	APPEARANCE
1	INNER RACE	Color of charcoal – shiny
	OUTER RACE	Gray – flaky to powdery metallic looking
	RETAINERS	Silvery Gray
2	INNER RACE	Gray – grainy
	OUTER RACE	Charcoal gray – satin finish
	RETAINERS	Gray – grainy
3	INNER RACE	Drak gray – glossy – good
	OUTER RACE	Gray glossy – slight graininess
	RETAINERS	Drak gray – glossy – good
4	INNER RACE	Charcoal gray – satin – some graininess
	OUTER RACE	Charcoal gray – flat (like flat paint)
	RETAINERS	Charcoal gray – shiny – some graininess

Assemblies were permitted to stay in the pressure chamber overnight, after flushing with Helium.

670-18 (2/64)

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SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	A.T.D.	673Y03-14	1200-41

DATA: SERVICE ITEM 3, SPECIMEN DETAILS AND HISTORY

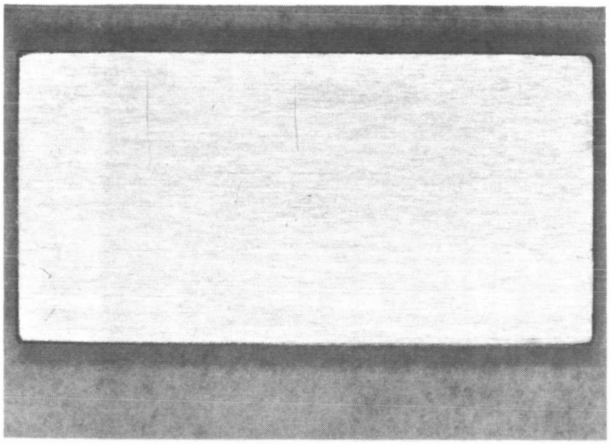

Figure 13–As Received, 3X.

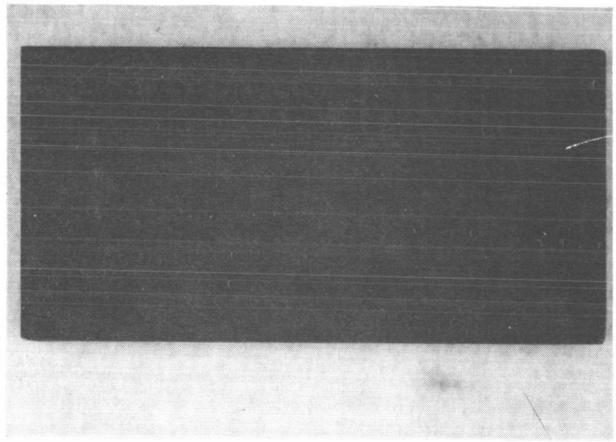

Figure 14–Post Plate, 3X.

Figures 8 and 9 show enlarged images of typical experimental specimens in the before and after plating conditions.

HISTORY AND PLATING DETAILS, TYPICAL

1. Material: AISI type 410 stainless steel
2. Substrate Hardness: Rockwell B-84
3. Dimensions: Length: 1.000 ± 0.003
   Width: 0.500 ± 0.003
   Thickness: 0.120 ± 0.003
4. Faces parallel $\pm$ 0.0003 total
5. Corners at $90^\circ \pm 30$ minutes
6. Face finish to be $\sqrt{16}$ ground finish
7. Specimen area:
$$A = [(1)\ (0.5)\ (2) + (0.5)\ (0.12)\ (2) + (1)\ (0.13)\ (2)]\ \text{ins.}^2$$
$$= (1 + 0.12 + 0.24)\ \text{ins.}^2$$
$$= 1.36 \text{ square inches}$$
8. Current per specimen at 12 ma per square inch
$$I = (1.36)\ (12)\ \text{ma} = 16.32 \text{ ma per specimen.}$$
9. Plate at 17.5 ma per specimen to allow for exposed specimen holder surfaces.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

Table 5

DATA: SERVICE ITEM 3, THICKNESS OF MoO_3 COMPLEX, OPTICAL MEASUREMENTS

Power Supply: See Figure 1
Total no. specimens plated at one time = 3
Total current each plating cycle = 52.5 ma
Voltage across system = 9 volts
pH before and after all experiments = 5.7
Specimen details: See Figure 13

SPECIMEN NO.	PLATE TIME MINUTES	PLATE THICKNESS	SPECIMEN NO.	PLATE TIME MINUTES	PLATE THICKNESS	SPECIMEN NO.	PLATE TIME MINUTES	PLATE THICKNESS
100	3	–	109	3	–	118	6	–
101	3	–	110	3	–	119	6	–
102	3	–	111	3	–	120	6	–
103	6	–	112	6	–	121	5	–
104	6	–	113	6	–	122	5	–
105	6	–	114	6	–	123	5	–
106	9	–	115	9	–	124	7	–
107	9	–	116	9	–	125	7	–
108	9	–	117	9	–	126	7	–

Note: In every case the thickness of the deposited MoO_3 complex was too small to measure optically.

670-18 (2/64)

W. G. Grenier — 2-14-64
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SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	A.T.D.	673Y03-14	1200-41

DATA: "Permascope" Type ESeJ4a Electronic Thickness Tester. Description of, by J.A. Munford, Fabrication Division.

"PERMASCOPE", TYPE ESeJ4a ELECTRONIC THICKNESS TESTER

The Permascope thickness tester, manufactured by Twin City Testing Corporation, provides a non-destructive means of measuring the thickness of both organic and non-magnetic metal coatings when applied to ferromagnetic substrates. This instrument has 4 thickness ranges enabling it to measure with microscopic accuracy coatings from 0.00" to 0.100". A constant pressure probe Type V11J4aS used in conjunction with scale I of the Permascope permits the measurement of coatings 0.000" to 0.0006" with the elimination of errors due to identation of the coating by the probe tips or curvature of the specimen.

When the probe is placed on the coating to be measured, the excitation circuit creates a magnetic field which penetrates the coating and closes itself inside the substrate. The magnitude of the magnetic flux depends on the thickness of the coating and influences the inductance of a pick-up coil. This coil is connected through a resistor to an AC source. The voltage drop in this coil, which depends on the inductance, and in turn on the thickness of the coating, is amplified and indicated on the instrument, which is graduated in thickness units.

The probe voltage and the amplifier are stabilized in order to maintain constant readings over long periods of time.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA:

Table 6
SERVICE ITEM 3, THICKNESS OF MoO_3 COMPLEX, PERMASCOPE MEASUREMENTS

Power Supply: See Figure 2
Number of specimens plated at one time = 2
Total plating current, each plating cycle = 35 ma
Voltage across plating cell = from 0.75 volts to 1.1 volts inclusive
Specimen details: See Figure 13
Odd numbered specimens will be retained for optical measurements

SPECIMEN NO.	PLATE TIME MINUTES	PLATE THICKNESS INCHES X 10^{-6}	APPEARANCE OF COATING AFTER PLATING
127	3	–	Shiny black → purple → blue → green
128	3	10–11	Shiny black → purple → blue → green
129	6	–	Uniform–black and shiny
130	6	31–32	Uniform–black and shiny
131	9	–	Uniform–black and shiny
132	9	50–50	Uniform–black and shiny
133	12	–	Shiny black–somewhat splotchy
134	12	81–84	Shiny black–somewhat splotchy
135	18	–	Shiny black–somewhat splotchy
136	18	96–96	Shiny black–somewhat splotchy
137	24	–	Shiny black–uniform
138	24	142–148	Shiny black–uniform
139	30	–	Shiny black–uniform
140	30	168-168	Shiny black–uniform

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: Table 7
SERVICE ITEM 3, THICKNESS OF MoO_3 COMPLEX, OPTICAL MEASUREMENTS

Odd numbered specimens only, from Table 6 given on preceding sheet.

SPECIMEN NO.	PLATE TIME MINUTES	READING NO.	PLATE THICKNESS INS. X 10^{-6}	SPECIMEN NO.	PLATE TIME MINUTES	READING NO.	PLATE THICKNESS INS. X 10^{-6}
127	3	–	–	135	18	1	145
						2	131
129	6	–	–			3	153
						4	105
131	9	1	105			AVERAGE–134	
		2	79				
		3	79	137	24	1	145
		4	95			2	121
		AVERAGE– 89				3	158
						4	171
133	12	1	111			AVERAGE–149	
		2	137				
		3	118	139	30	1	163
		4	116			2	168
		AVERAGE–120				3	147
						4	179
						AVERAGE–166	

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

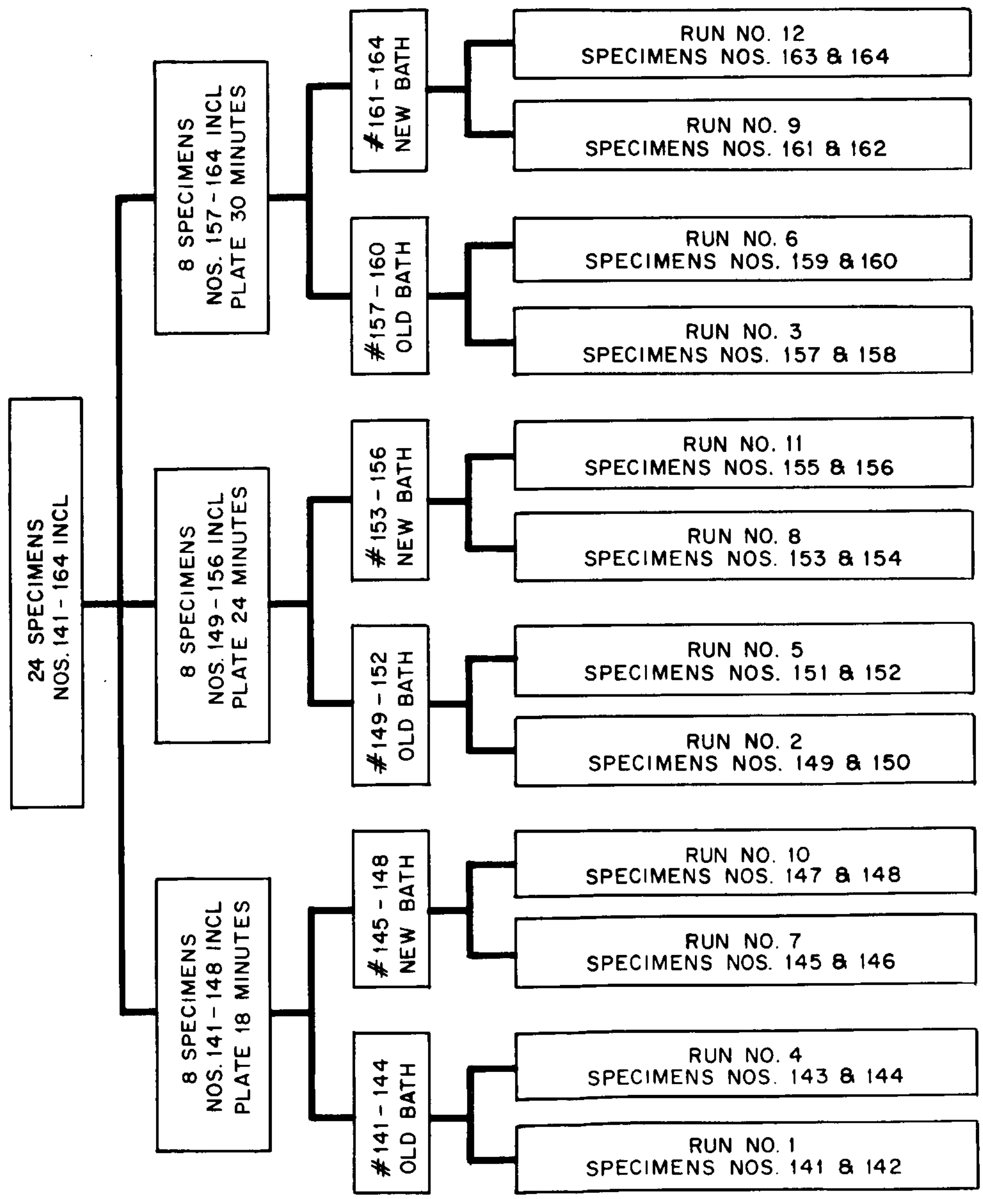
SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA:

Table 8
SERVICE ITEM NO. 3
FLOW CHART OF SPECIMEN PLATING CONDITIONS, SELECTION

For specimen details see Figure 13.



670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	A.T.D.	673Y03-14	1200-41

DATA: Table 9
"PERMASCOPE" THICKNESS MEASUREMENTS BY J. A. MUNFORD

THICKNESS OF COATING IN MILLIONTHS OF AN INCH
ON DESIGNATED SPECIMENS FACES

READING NO.	ONE FACE OF SPECIMEN							OTHER FACE OF SPECIMEN							OVERALL AVERAGE
	1	2	3	4	5	6	AVG.	1	2	3	4	5	6	AVG.	
SPECIMEN NO.	18 MINUTE PLATE – OLD BATH														
141	108	110	108	111	110	108	109	108	108	111	110	110	107	109	109
142	88	85	88	88	90	88	88	92	97	95	98	92	94	95	91
143	112	111	112	113	112	110	112	112	110	109	109	111	109	110	111
144	125	125	127	130	125	127	127	122	121	121	123	128	128	124	125
	18 MINUTE PLATE – NEW BATH														
145	160	160	165	163	160	159	161	142	150	148	148	155	155	150	155
146	130	130	130	129	129	130	130	125	124	128	123	125	124	125	127
147	142	142	140	140	139	140	140	142	145	145	139	140	140	142	141
148	132	138	133	138	131	133	134	135	134	131	129	130	131	132	133
	24 MINUTE PLATE – OLD BATH														
149	232	232	235	227	228	220	229	228	230	230	225	230	232	229	229
150	SURFACE UNEVEN – NO READINGS POSSIBLE													–	–
151	220	222	230	228	230	232	224	235	240	238	235	240	230	236	232
152	170	163	168	168	170	175	169	172	170	174	178	176	175	174	172
	24 MINUTE PLATE – NEW BATH														
153	195	195	190	192	196	197	194	185	189	195	195	198	192	194	193
*153-1	180	178	175	180	172	178	177	178	181	190	188	170	180	182	180
154	180	183	180	182	182	180	181	190	178	170	185	188	183	182	182
*154-1	170	170	170	172	175	172	171	175	170	170	172	175	177	173	172
155	160	163	165	162	162	172	164	172	162	170	172	175	170	170	163
156	190	190	188	192	192	196	191	197	190	195	192	189	193	193	192

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	A.T.D.	673Y03-14	1200-41

DATA: Table 9 (Continued)

THICKNESS OF COATING IN MILLIONTHS OF AN INCH
ON DESIGNATED SPECIMENS FACES

READING NO. / SPECIMEN NO.	ONE FACE OF SPECIMEN 1	2	3	4	5	6	AVG.	OTHER FACE OF SPECIMEN 1	2	3	4	5	6	AVG.	OVERALL AVERAGE
	30 MINUTE PLATE – OLD BATH														
157	162	166	168	163	167	165	165	160	160	158	155	158	158	158	162
158	135	139	140	136	142	150	140	152	158	149	149	142	160	152	146
159	168	170	163	168	170	171	168	185	178	178	185	182	180	181	175
160	185	183	188	190	188	182	186	179	183	180	178	185	183	181	184
	30 MINUTE PLATE – NEW BATH														
161	210	212	212	215	218	211	213	218	222	218	218	214	209	217	215
162	225	235	231	229	222	231	229	232	234	225	222	230	230	229	229
163	230	228	230	232	232	232	231	230	231	228	230	233	230	230	231
164	225	225	228	228	223	219	225	228	228	230	230	227	232	229	227

*Due to the streaked appearance of specimen numbers 153 and 154 after plating, these specimens were plated under identical conditions with the exception that the pre-plate activation bath was first cleansed of all foreign matter.

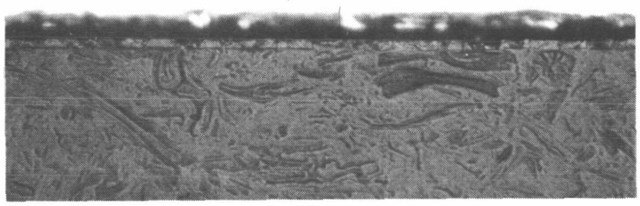
670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

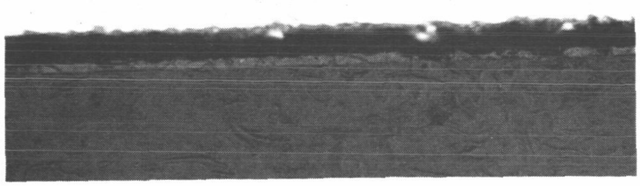
ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: SERVICE ITEM NO. 3
PHOTOMICROGRAPHS, AS POLISHED SPECIMENS



250X

Figure 15–Specimen No. 153.



250X

Figure 16–Specimen No. 162.

Figures 15 and 16 point up some of the problems encountered when determining plate thickness by optical methods. The two specimens shown are typical of five such, which were mounted in green bakelite and are presented in the as polished condition. Note that in both photomicrographs the following is apparant: (1) The edge of the substrate is rounded; (2) The polished surface of the substrate is at a different level than the bakelite; (3) The bakelite shrunk away from the specimen and the plated MoO_3 complex adhered more tightly to it than to the substrate.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	A.T.D.	673Y03-14	1200-41

DATA:

Table 10
THICKNESS OF MoO_3 COMPLEX, OPTICAL MEASUREMENTS

THICKNESS OF COATING IN MILLIONTHS OF AN INCH
ON DESIGNATED SPECIMEN FACES

READING NO.	ONE FACE OF SPECIMEN							OTHER FACE OF SPECIMEN					OVERALL AVERAGE
	1	2	3	4	5	6	AVG.	1	2	3	4	5	
SPECIMEN NO.	18 MINUTE PLATE – OLD BATH												
143	118	131	116	147	126	128	119	103	113	131	103	112	120
	24 MINUTE PLATE – NEW BATH												
153	187	187	197	203	197	194	205	191	203	184	194	200	185
156	191	194	203	197	194	196	165	187	194	187	168	180	188
	30 MINUTE PLATE – OLD BATH												
159	187	187	187	150	191	180	189	184	200	215	205	199	190
	30 MINUTE PLATE – NEW BATH												
162	321	218	228	218	234	244	231	239	231	276	228	241	242

Measurements in this table were made on specimens mounted in green bakelite, as those shown in Figures 15 & 16, sheet 51.

Instrument used: Bausch & Lomb Research Metallograph equipped with a Filar micrometer eyepiece and the 50 X objective lens.

1 filar unit = 2.62×10^{-6} inches.

670-18 (2/64)

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: Table 11
SERVICE ITEM 3, PLATING MoO_3 COMPLEX, SYNOPSIS, SPECIMENS 141-164

RUN NO.	SPECIMEN NO.	PLATE TIME MINS.	BATH TYPE	AVERAGE PLATE THICKNESS INS. X 10^{-6}		PLATING APPEARANCE
				PERMASCOPE	OPTICAL	
1	141	18	OLD	109		Shiny black, uniform
1	142	18		91		Shiny black, uniform
2	149	24		229		Dull black, uniform
2	150	24				Dull black, uniform
3	157	30		162		Shiny black, uniform
3	158	30		146		Shiny black, uniform
4	143	18		111	120***	Shiny black, uniform
4	144	18		125		Shiny black, one face is splotchy
5	151	24		232		Shiny black, uniform
5	152	24		172		Shiny black, uniform
6	159	30		175	190***	Shiny black, uniform
6	160	30		184		Shiny black, uniform
7	145	18	NEW	155		Shiny black, uniform
7	146	18		127		Shiny black, uniform
8	153	24		193	185***	Shiny black, Streaked**
8	154	24		182		Shiny black, Streaked**
9	161	30		215		Shiny black, uniform
9	162	30		229	242***	Shiny black, uniform

670-18 (2/64)

W. G. Grenier 2-14-64
(Signature) (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: Table 11 (Continued)

RUN NO.	SPECIMEN NO.	PLATE TIME MINS.	BATH TYPE	AVERAGE PLATE THICKNESS INS. X 10^{-6}		PLATING APPEARANCE
				PERMASCOPE	OPTICAL	
10	147	18	NEW	141		Shiny black, uniform
10	148	18		133		Shiny black, uniform
11	155	24		163		Shiny black, uniform
11	156	24		192	188***	Shiny black, uniform
12	163	30		231		Shiny black, spotted, like water spots
12	164	30		227		Shiny black, spotted, like water spots
13*	153-1	24		180		Shiny black, uniform
13	154-1	24		172		Shiny black, uniform

NOTES:

*Not included in Table No. 8, Flow Chart of Specimens.

**Streaks probably due to thin film of Glyptol on surface of activation bath, some of which may have adhered to specimen.

***Optical thickness readings taken on specimens mounted in green bakelite, as shown in Figures 16 and 17.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y04-14	1200-41

DATA: SERVICE NO. 3
ELECTRODEPOSITION OF MoO_3 COMPLEX, OLD BATH – TABLE NO. 9

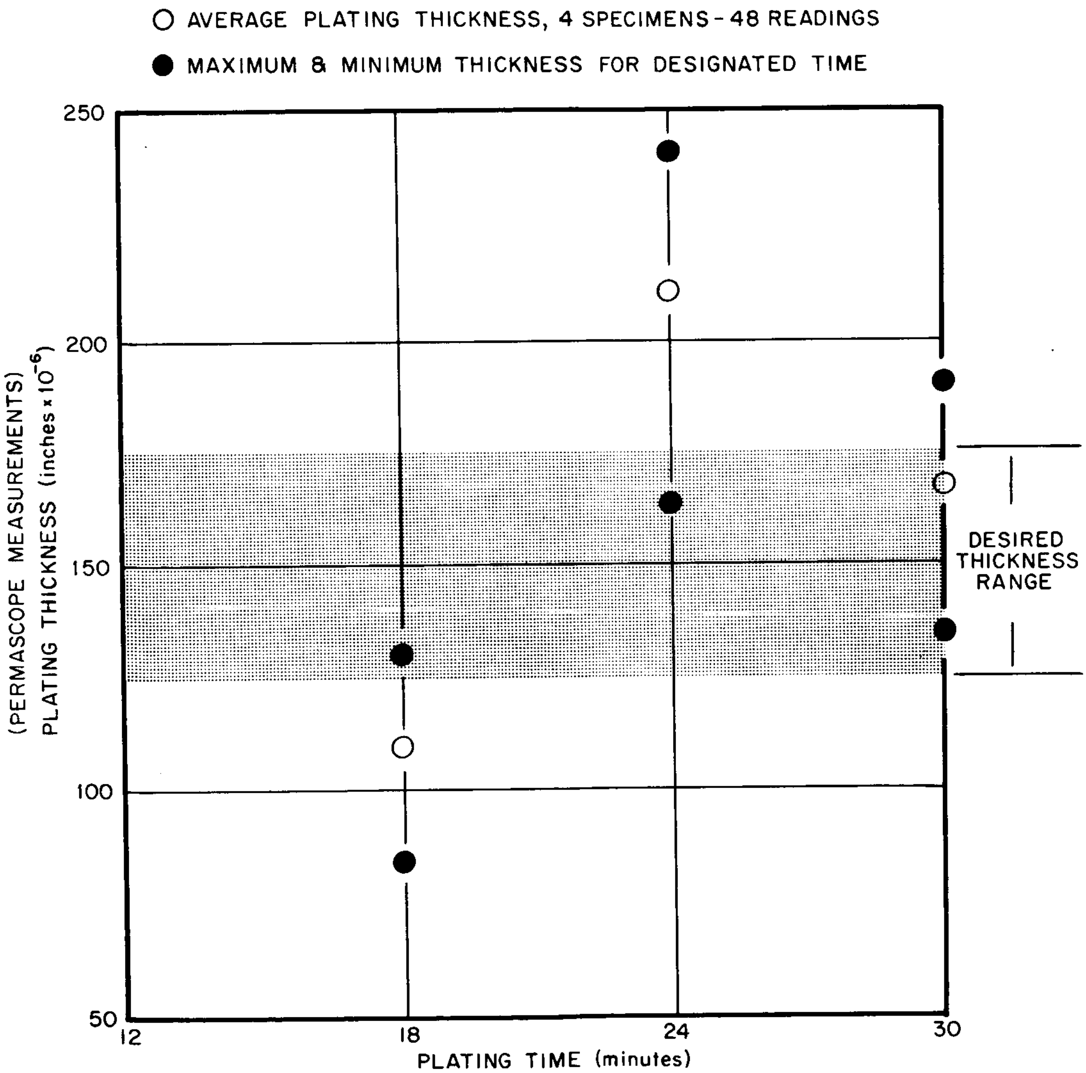


Figure 18–Electrodeposition Time Compared to Resultant Thickness.

Note: Old bath is same as that used for Figure 17.

W. G. Grenier — *(Signature)*
2-14-64 — *(Date)*

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA: SERVICE NO. 3
ELECTRODEPOSITION OF MoO_3 COMPLEX ON 410 S.S., AS TABLE NO. 6

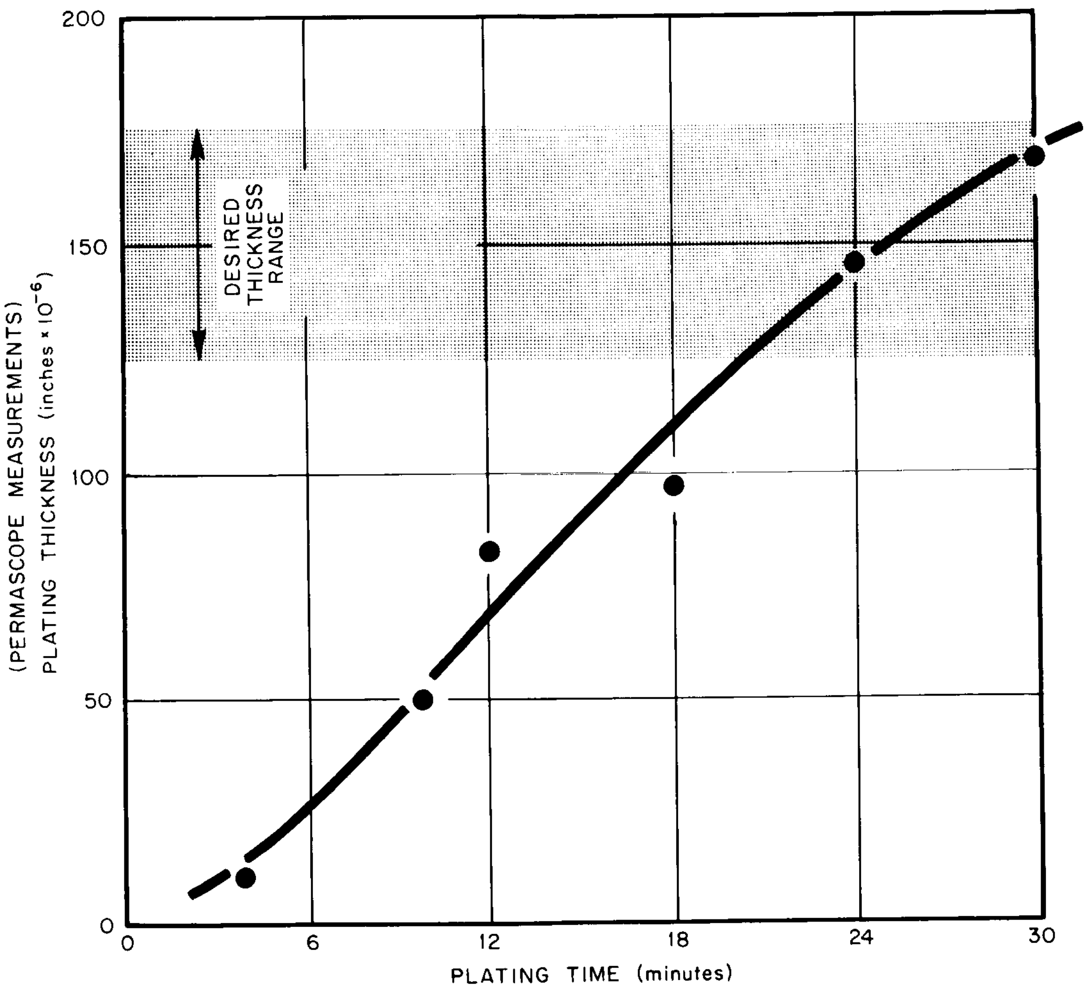


Figure 17–Electrodeposition Time Compared to Resultant Thickness.

Preliminary data where each point represents the average plating thickness on one specimen. These findings suggested the approach shown in Table 8.

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT
STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

DATA:
SERVICE NO. 3
ELECTRODEPOSITION OF MoO_3 COMPLEX, NEW BATH – TABLE NO. 9

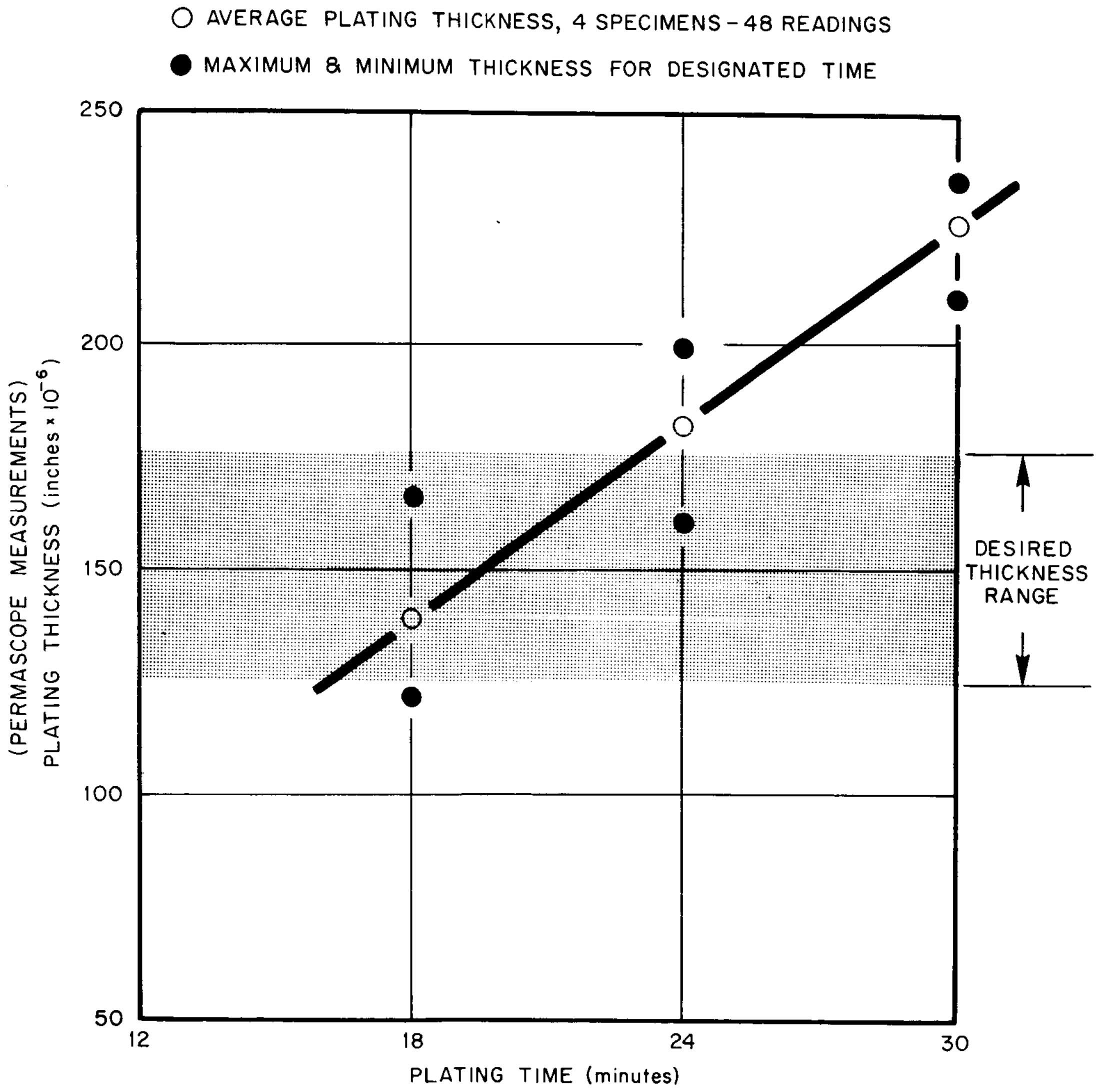


Figure 19–Electrodeposition Time Compared to Resultant Thickness.
(New Bath)

670-18 (2/64)

W. G. Grenier (Signature) 2-14-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

CONCLUSIONS

A great deal of work has been performed on this Mos_2 "in Situ" program and it has been reported in a number of S&MA Section Service Reports – dates 9-11-62, 11-4-63, (TR-1200 29) 10-24-62 and 2-12-64 – and a T.N.

The work performed an electrodeposition has been sufficient to establish a controllable process. Thickness can be controlled at a nominal of 150 microinches to ± 25 microinches. The thickness that can be deposited approaches a maximum of 250 microinches and a minimum that can not be measured by our laboratory techniques. Careful laboratory techniques are required for this close control and it is felt that ± 50 microinches can be attained on a production bases. The process developed by the laboratory will produce a good adherent and reproducible product.

The use of a non-destructive method of measuring film thickness (Permascope tester – using either magnetic substrate and non-magnetic film or vice versa) is shown to be more reliable and quicker than the optical method used by the laboratory (destructive method).

Conclusions:

1. The film (MoS_2 in Situ) can be controlled to a thickness of ±25 microinches with a nominal of 150 microinches under tightly controlled laboratory conditions.

670–15

(Signature) 3-5-64 (Date)

SERVICE REPORT

STRUCTURAL AND MECHANICAL APPLICATIONS SECTION

ORIGINATOR	PROJECT	JOB ORDER NUMBER	REQUEST NUMBER
C. E. Vest	MoS_2 In Situ	673Y03-14	1200-41

CONCLUSIONS

2. The Permascope thickness tests is suitable and reliable for measuring the in Situ MoS_2 film in this range and is more desirable than the optical (destructive method).

C. E. Vest (Signature) 3-5-64 (Date)